Comparison of Two Potassium-Filled Gas-Controlled Heat Pipes

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Abstract

Calibration by comparison of platinum resistance thermometers and thermocouples requires transfer media capable of providing very good short-term temperature uniformity and temperature stability over a wide temperature range. This paper describes and compares the performance of two potassium-filled gas-controlled heat pipes (GCHP) for operation over the range from 420 °C to 900 °C. One of the heat pipes has been in operation for more than 10 years having been operated at temperature for thousands of hours, while the other was commissioned in 2010 following recently developed improvements to both the design, assembly, and filling processes. It was found that the two devices, despite differences in age, structure, number of wells, and filling processes, realized the same temperatures within the measurement uncertainty. The results show that the potassium-filled GCHP provides a durable and high-quality transfer medium for performing thermometer calibrations with very low uncertainties, over the difficult high-temperature range from 420 °C to 900 °C.

Keywords: Gas-controlled heat pipe, Pressure control, Thermocouple calibration, Thermometer calibration

1 Introduction

Calibration by comparison of platinum resistance thermometers and thermocouples requires devices capable of providing uniform and stable temperatures. Usually laboratories use furnaces with an equalizer block to generate temperatures above 550 °C. The best stability and uniformity of typical furnaces, used in calibration for comparison, is some tenths of degree. These contributions usually are the biggest source of uncertainty in the budget to estimate the uncertainty of calibration of temperature probes. The need of customers is to reduce the uncertainty of calibration of thermometers and thermocouples between 420 °C and 900 °C, in order to improve their production processes. In this temperature range, there is a general lack of suitable high temperature media: oil baths limited to 250 °C to 300 °C, salt baths limited to 550 °C, and metal blocks have uniformity and response time problems. Moreover, such devices have a long response time in terms of reaching stability after a temperature change, required to execute several temperature calibration points.

Gas-controlled heat pipes (GCHPs) have been used for studies on vapor-pressure scales and thermometer non-uniqueness [1, 2, 3, 4, 5, 6] since they can achieve stabilities on the order of a few millikelvin over wide temperature ranges and spatial temperature uniformity of the same order. Such devices can therefore be efficient systems responding to calibration requirements [7].

Under a commercial-research contract for a customer working as a calibration accredited laboratory, a gas-controlled heat pipe (GCHP) has been manufactured under an INRiM design following the recent advances in the design of these devices. Using a GCHP, the performance of stability and uniformity improves by an order of magnitude. The thermodynamic relationship linking temperature and pressure in a fluid at a phase transition, guarantees to reach a quick temperature change. This feature can shorten the time required for reaching different calibration points, thus saving funds in terms of person time and power consumption, with respect to the reported “traditional” baths, blocks, and furnaces.

The paper reports the features of the complete device, including the auxiliary systems. A complete description of the GCHP, the associated pressure line, cooling line together with the description of the filling, and experimental procedures adopted for the purpose is given.

Together with the GCHP, the complete system, including heating equipment, cooling lines, a pressure line, and safety systems, was assembled and delivered.

This is the first time a GCHP-based system is made to be used outside scientific or NMI laboratories. For this purpose, the whole system was designed to allow easy use, commercially available and replaceable parts, lower costs, and long lasting use capabilities, together with complete care on operator safety.

To guarantee traceability and complete operational characterization, a direct comparison between the customized GCHP being characterized and the potassium GCHP operating at INRiM as a calibration facility since more than a decade was performed. This work has not only allowed a knowledge of the operational aspects of the customized GCHP, but it has contributed to a direct comparison, currently the only one, with a GCHP used for the calibration of thermometers.

The characterization of the customized gas-controlled heat pipe (GCHP) with seven wells was carried out at the “Laboratorio di Termometria Industriale per Contatto” of the Thermodynamics Division of INRiM in the period between September 2010 and December 2010. The tests were carried out at different temperatures and involved the study of the temperature stability inside the thermowells, and the temperature uniformity along the vertical axis of the GCHP and among the seven thermowells.

The work presented here reports the stability and uniformity tests on the GCHP and the results of the comparison.

This is the first comparison ever made between two of those devices used for calibration. Results give information about the performance improvements for the new modified GCHP compared to the GCHP used at INRIM built ten years ago, and to evaluate the effects of the use of the GCHP of INRIM after ten years of activity of calibration of thermometers.

The results of the comparison, demonstrating both the comparability and repeatability of the GCHPs, together with the thermal quality in terms of stability and uniformity, open the perspective of surpassing the prototype phase in the development of such devices. A possible interest for commercializing them as devices for temperature sensor calibrations will then be investigated together with interested companies.

2 Experimental Setup

2.1 The GCHP

GCHPs realize the potassium liquid-vapor phase transition over the whole measurement volume. Since equilibrium is constantly maintained by means of appropriate heating and cooling, a temperature change immediately occurs at any pressure change. Therefore, as is well known, the temperature inside the GCHP can be controlled by controlling the pressure of the phase transition. A vapor/gas interface is realized with helium as the buffer control gas.

The heat pipes used in this project are both made of Inconel in the form of a cylindrical envelope closed on both ends by discs of the same material (Fig. 1) . The dimensions of the GCHPs used in this study are: 455 mm height, 114 mm diameter, and 3 mm thickness. From previous experience, the heat pipe is filled with 200 g of potassium of 99.95 % purity to ensure correct operation. The pressure control system for the new GCHPs is equipped with a modified commercial pressure controller provided with a lower pressure limit, down to 1000 Pa, according to the temperature range requested by the customer, taking into account the pressure-temperature curve of the potassium vapor-liquid phase transition (Fig. 2).

The power required to maintain the liquid/vapor phase transition inside the GCHP is provided by a single-zone furnace with side heaters connected to a Variac and a transformer: 1.2 kW of electric heating power are required for operating the GCHP at 900 °C. In order to improve the performance, the new GCHP is provided with seven thermometer wells, open at the top and closed at the bottom where they are immersed in the GCHP: six thermometer wells are placed on a circle and one is added in the center to the new heat pipe. This new configuration was designed to improve the uniformity between the thermometer wells, placing the standards (PRTs or thermocouples) in the central position, having at the same time six wells available to calibrate temperature probes (in the INRIM GCHP there are only five wells).

To improve the performance of the stability and axial uniformity, the new GCHP has a different capillary structure with respect to the old one: the wicks are machined with an angle of 90° toward the top part and approximately 45° towards the bottom, to allow better permanence of the drops during condensation. A mesh is added to both GCHPs, and the surfaces are not treated to induce condensation.

Two separate cooling lines are connected to the older INRiM GCHP. The first water-cooling line is used to cool the furnace for protecting the laboratory and the operators from the heat. The second line provides the necessary cooling to the GCHP chimney, in order to keep it working properly; it represents the fundamental cooling that causes the vapor to condense back to the liquid state and return to the bottom of the GCHP. This second cooling line keeps water flowing also on the top of the thermometer wells to increase the amount of fluid condensation around the measuring zone. The amount of water flowing in both cooling lines is separately controlled, in order to guarantee the best heating/cooling ratios and thus achieve the best operating capabilities of the heat pipe. For balancing between the costs of the apparatus and the application and target uncertainties, the custom new GCHP is provided with a single cooling line. The initial condensation along the thermometer wells is stimulated by inserting a metal rod at room temperature.

2.2 Pressure Line

A pressure line has been assembled to be associated with this GCHP. It is based on several 6 mm stainless steel tubes and connectors, manual valves, a vacuum pump, a 50 L buffer volume, and a gas tank for the helium that is used as a controlling gas. The commercial pressure controller is connected to the pressure line following the manufacturer's instructions and some custom adaptations. The pressure line has been vacuum tested and packed ready to be delivered together with the GCHP.

For the characterization and comparison, the new GCHP has been connected to the same pressure line of the GCHP operating at INRiM, through a console that allows multiple GCHP connections for research and calibration purposes. This line uses a commercial Druck DPI 510 controller, equipped with two pressure transducers for operating in two pressure ranges. The first sensor, the gauge type, has a full scale of 35 kPa, while the second, the absolute type, has a full scale of 300 kPa.

2.3 Measurement Line

The measurement line that has been set-up at the INRiM laboratory consists of the following instruments:

- a HTSPRT, Model R800-3, made by the manufacturer CHINO, having the following characteristics: nominal resistance of 2.5 Ω at the triple point of water; mounting four terminals of platinum with insulation pipe and protective sheath, transparent frosted quartz long stem; operating range from 0.01 °C to 961.78 °C;
- precision AC thermometry bridge, Model F18, manufacturer ASL, with maximum uncertainty of the resistance ratio of 1 ppm and equipped with a 10 Ω external reference resistance, and Tinsley Model 5685, calibrated at INRiM having 3 ppm relative uncertainty.

2.4 Filling Procedure

The GCHP has been filled at INRiM following a well-defined procedure, adopted and defined at INRiM to guarantee the working fluid purity to be preserved by accidental contact with air during the transfer from the sealed containers to the GCHP. The procedures allow transfer of the potassium from the sealed bulbs; it is delivered into the GCHP with no contact with ambient air in order to avoid oxidation. It is based on a dedicated pressurized line, filled with argon and equipped with a heater to melt the potassium and pour it into the GCHP through its chimney. The bulbs are inserted in the pressure line in a special cylinder that allows to break them softly, heat them, and wait for the potassium to flow inside the GCHP. All of this procedure is done with no air contaminating the melted metal. The GCHP with the new design is equipped with a 40 mm diameter pressure junction on the top, to allow easy filling and possible change of the working fluid.

2.5 Experimental Procedures

The temperature stability measurements and the vertical gradient evaluations were carried out mainly in the central well of the customized GCHP while separate runs of measurements in each well were made to evaluate the radial temperature gradient .The points that have been subject to measurements were set at pressures of 1 kPa, 37 kPa, 90 kPa, 200 kPa, and 320 kPa, corresponding to temperatures near 420 °C, 660 °C, 750 °C, 840 °C, and 900 °C, respectively.

3 Results

The temperature stability and uniformity evaluations are reported here only for the custom GCHP, since the one already in use by INRiM is well characterized, as it is part of defined INRiM calibration procedures [8].

3.1 Stability Test

The stability tests involved the study of the variation with time of the temperature inside the GCHP central thermowell. For these tests, measurements were carried out at 420 °C, 660 ° C, 750 ° C, 40 °C, and 900 ° C in observation periods of 60 minutes, comparable to the duration of the calibration procedures for which it is provided the use of the GCHP, sampling the temperature every minute.

The results of the stability tests at temperatures of about 660 °C (37000 Pa) are shown as an example in Fig. 3; the results of all temperature stabilities are summarized in Table 1.

3.2 Temperature Vertical Gradient

Uniformity tests were conducted along the central well of the GCHP, by performing measurements of the temperature changes positioning the HTSPRT initially at the bottom of the well and then extracting it 2 cm by 2 cm up to the point where the temperature drops significantly. The results of the axial uniformity at all temperatures (420 °C, 660 ° C, 750 ° C, 840 ° C, and 900 ° C) are shown in Fig. 4.

The axial uniformity stays within ±5 mK along about 15 cm in all thermowells.

3.3 Radial Uniformity Among the Thermometer Wells

The tests of uniformity between the wells of the GCHP were made by comparing the central well with all of the six radial symmetric wells. Figure 5 shows trends of the uniformity at temperatures of about 900 ° C (320 000 Pa) with reference to Fig. 6 for the nomenclature of thermowells.

Conducting the test of uniformity, the results obtained for each temperature are show in Table 1.

At low temperatures the radial uniformity is within 0.03 °C, but the best radial uniformity (within 0.01 °C) is obtained at higher temperatures, due to an increase in performance of the pressure control system.

3.4 Comparison Between the Two GCHPs

Test results are compared with the performance of the INRIM heat pipe.

Results of stability tests in both systems show that the performances are substantially the same.

An additional thermowell placed in the center of the new heat pipe led to an improvement of the radial uniformity above 750 °C compared to the old HP.

Also, axial uniformity is improved in the whole range of temperature, due to the different capillary structures with respect to the old one. This is an important improvement, because the GCHP can also be used to perform the tests of homogeneity of the thermocouples in calibration [11].

A comparison of this GCHP with respect to the one already operating at INRiM has been performed. Except for a similar comparison carried out for research purposes between two sodium GCHPs, one of INRiM and the other of CNAM (Conservatoire national des arts et métiers - Paris) [9, 10], this is the first time that such a comparison takes place, between devices used for calibration purposes. Measurements have been made connecting the customer GCHP to the same pressure line of the GCHP in use, thus operating at the same temperature.

The same HTSPRT has been used, and the measurements were carried out at the same pressures as the uniformity and stability tests.

It has to be noticed that the presence of the two GCHPs together did not disturb the control capabilities; thus, it did not affect the temperature performances in terms of stability. Measurements were made moving the HTSPRT initially placed in the central well of the customer GCHP, then extracted and rapidly inserted into the well of the INRiM GCHP that usually hosts the standard thermometer of the laboratory. The operation was performed repeatedly, with the necessary waiting times for the thermometer and the pressure inside the GCHP to restore the thermal stability, for a total of five measurements overall.

Figure 7 shows the comparison between the two GCHPs at a temperature of about 600 °C (37 000 Pa).

The results of the comparison show a good level of confidence, showing that the two devices reproduce the same temperature, at the same pressure, within a hundredth degree.

4 Conclusion

Tests have been carried out investigating the stability and uniformity performance of HPs. Results show that the new design of the capillarity structure realized in the customer's HP, has led to an extension of its axial uniformity area.

The addition of a central thermowell in the customer's HP has reduced the gradient between thermowells in the range above 750 °C.

Results of a comparison between two heat pipes have shown that after ten years of calibration activity, the system of INRIM GCHP maintains a reproducibility very similar to the new GCHP, within a hundredth of a degree.

The potassium used to fill the new GCHP comes from the same batch and same provider of the one used for the older one: such a result indicates that during the ten years of operation, no contamination was released by the Inconel walls, nor oxidation occurred due to even minimal leakages. The potassium-filled GCHP together with the power equipment, cooling lines, the pressure line, and controller is now fully characterised and ready to be shipped to the customer and easily assembled to start the calibration activities.

Acknowledgment

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Table 1 Summary of results of the stability and radial uniformity test at all temperature

Temperature (°C)	Standard deviation[a] (K)	ΔT[b] (K)
420	0.003	≤ 0.03
660	0.002	≤ 0.03
750	0.002	≤ 0.03
840	0.002	≤ 0.01
900	0.002	≤ 0.01

[a] Stability during 1 hour of measurement
[b] Radial uniformity among the thermometer wells, difference between all thermowells

Figure Captions

Fig. 1 Photo of the customized GCHP

Fig. 2 Relationship between pressure and temperature of the potassium in saturation conditions (logarithmic scale)

Fig. 3 Stability test of the custom GCHP at temperature of about 660 °C

Fig. 4 Axial uniformity of central well of the heat pipe customized GCHP at all temperatures

Fig. 5 Uniformity between the wells of the custom GCHP at temperature of about 900 °C

Fig. 6 Photo of GCHP under comparison with thermometer wells numbering

Fig. 7 Comparison between the two GCHPs at temperature of about 660 °C

Fig. 1

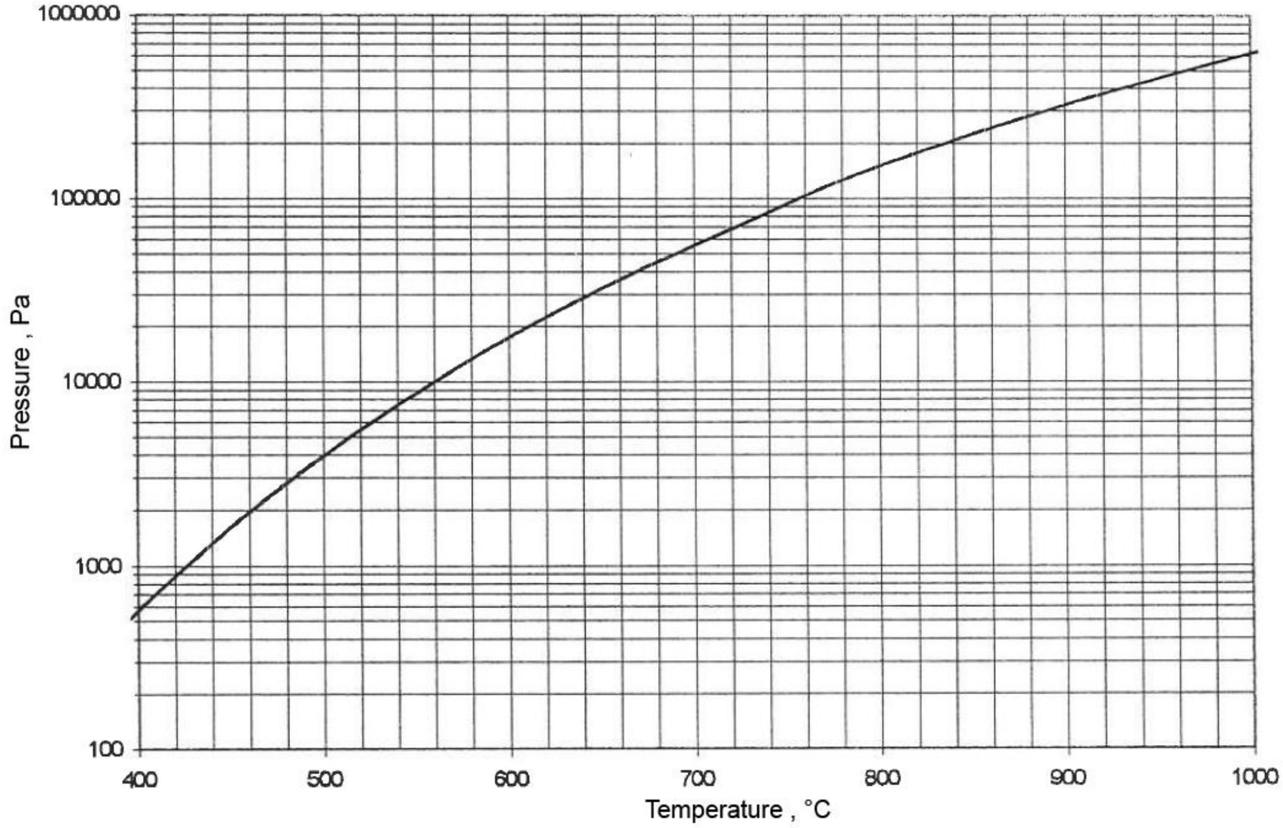

1000000
100000
10000
1000
100
Pressure , Pa
400
500
600
700
800
900
1000
Temperature , °C

Fig. 2

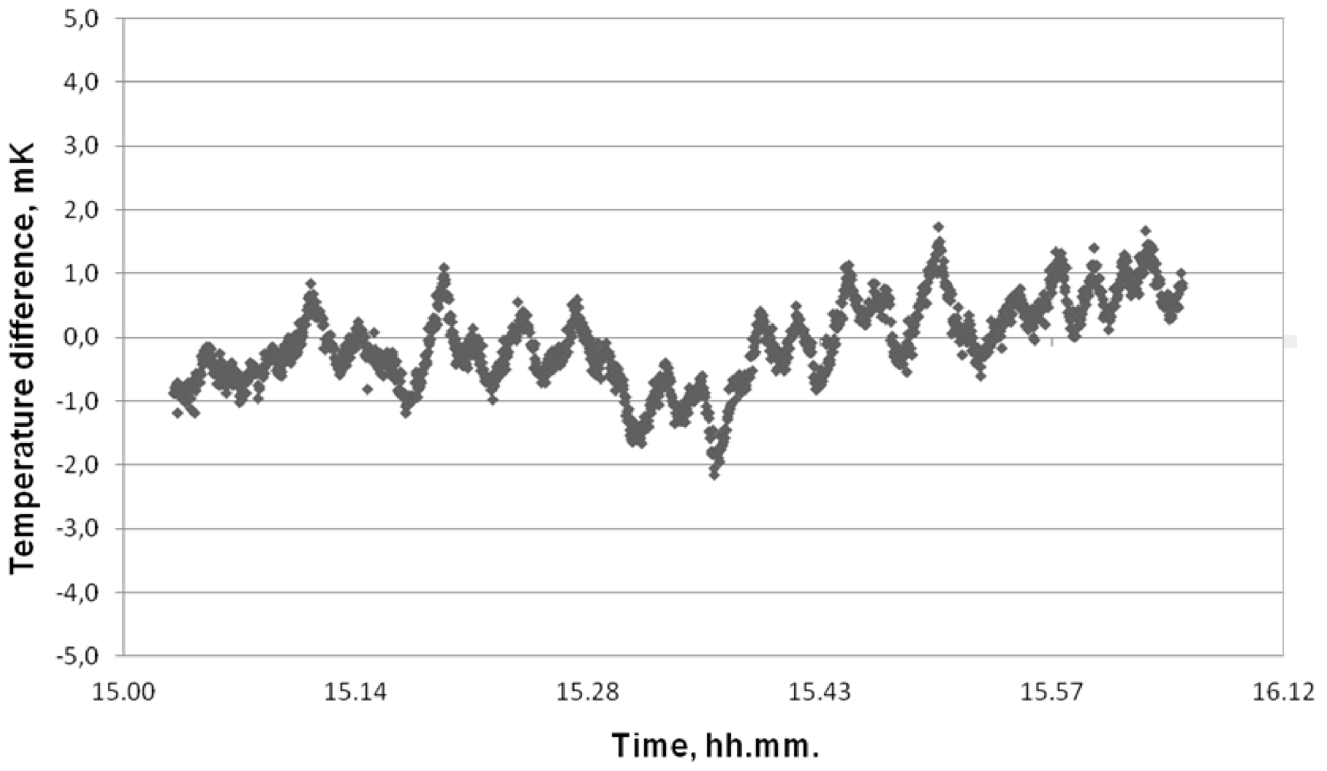

5,0
4,0
3,0
2,0
1,0
0,0
-1,0
-2,0
-3,0
-4,0
-5,0
Temperature difference, mK
15.00
15.14
15.28
15.43
15.57
16.12
Time, hh.mm.

Fig. 3

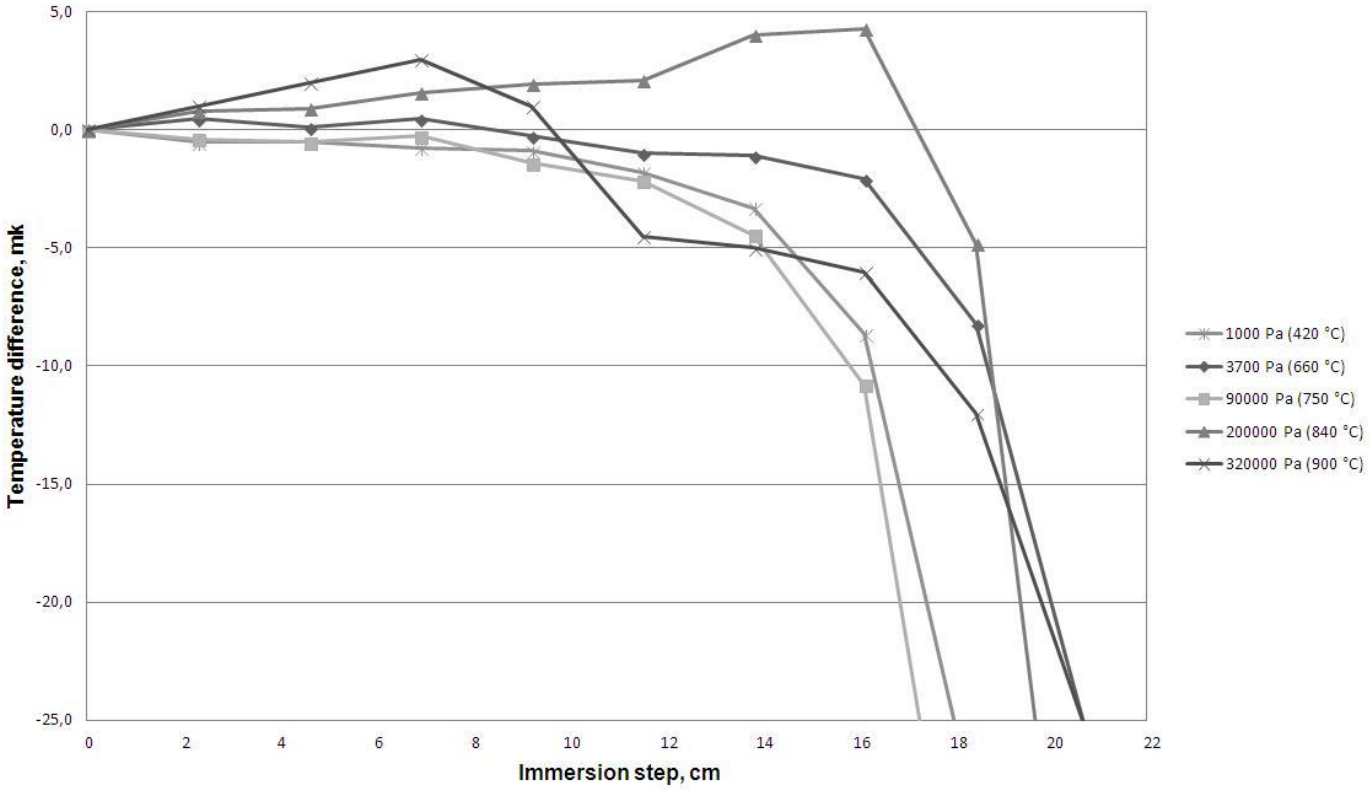

5,0
0,0
-5,0
-10,0
-15,0
-20,0
-25,0
Temperature difference, mk
0
2
4
6
8
10
12
14
16
18
20
22
Immersion step, cm
1000 Pa (420 °C)
3700 Pa (660 °C)
90000 Pa (750 °C)
200000 Pa (840 °C)
320000 Pa (900 °C)

Fig. 4

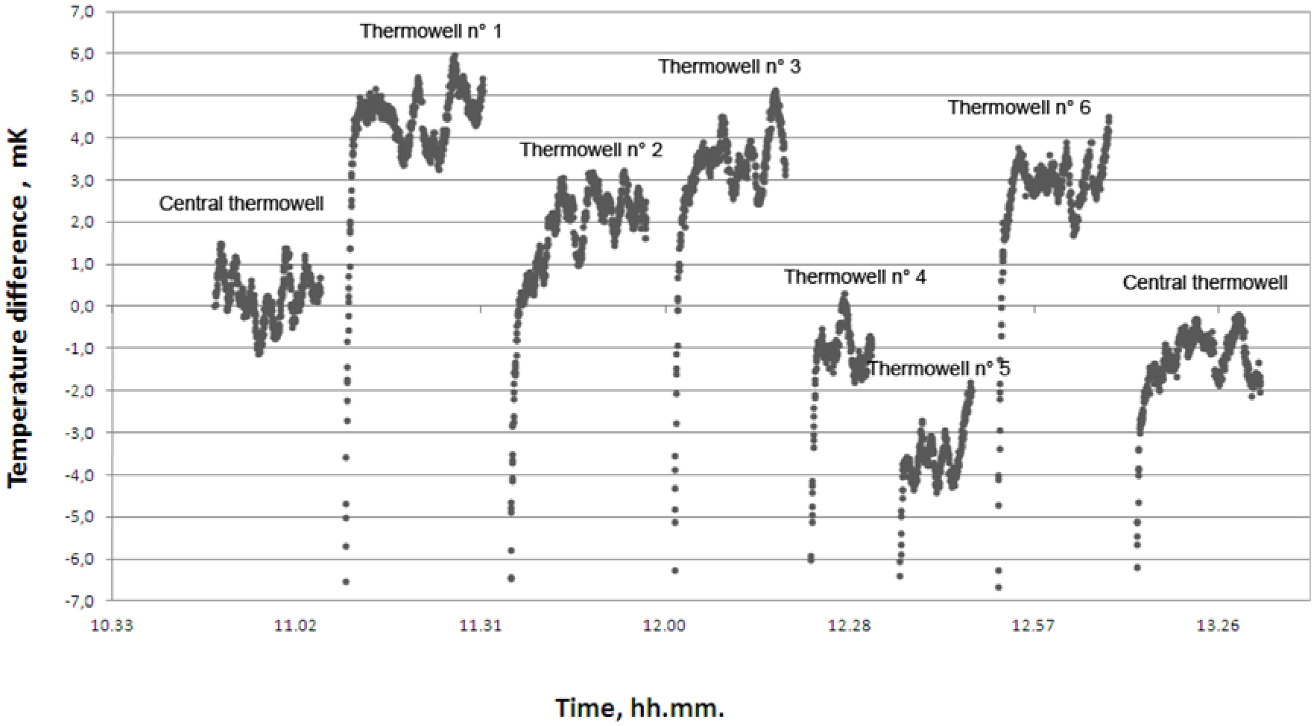

Thermowell n° 1
Thermowell n° 3
Thermowell n° 6
Thermowell n° 2
Central thermowell
Thermowell n° 4
Thermowell n° 5
Central thermowell
Temperature difference , mK
7,0
6,0
5,0
4,0
3,0
2,0
1,0
0,0
-1,0
-2,0
-3,0
-4,0
-5,0
-6,0
-7,0
10.33
11.02
11.31
12.00
12.28
12.57
13.26
Time, hh.mm.

Fig. 5

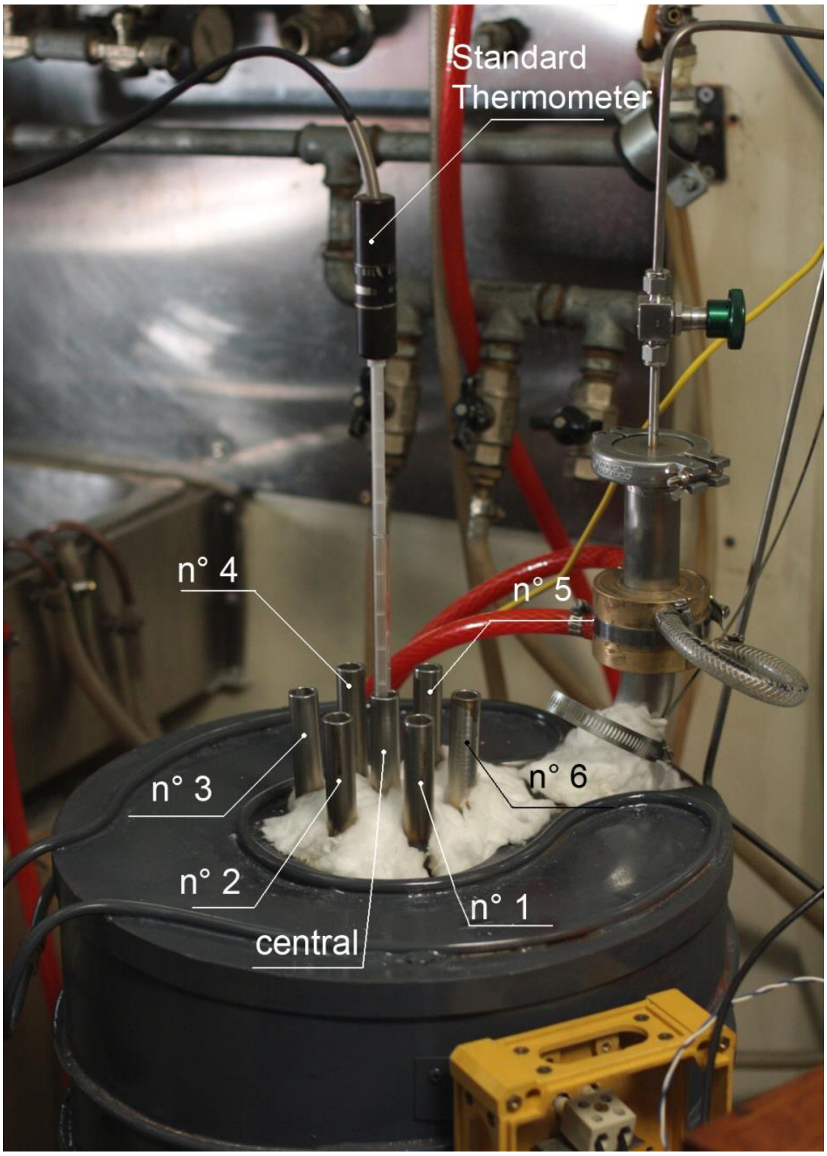
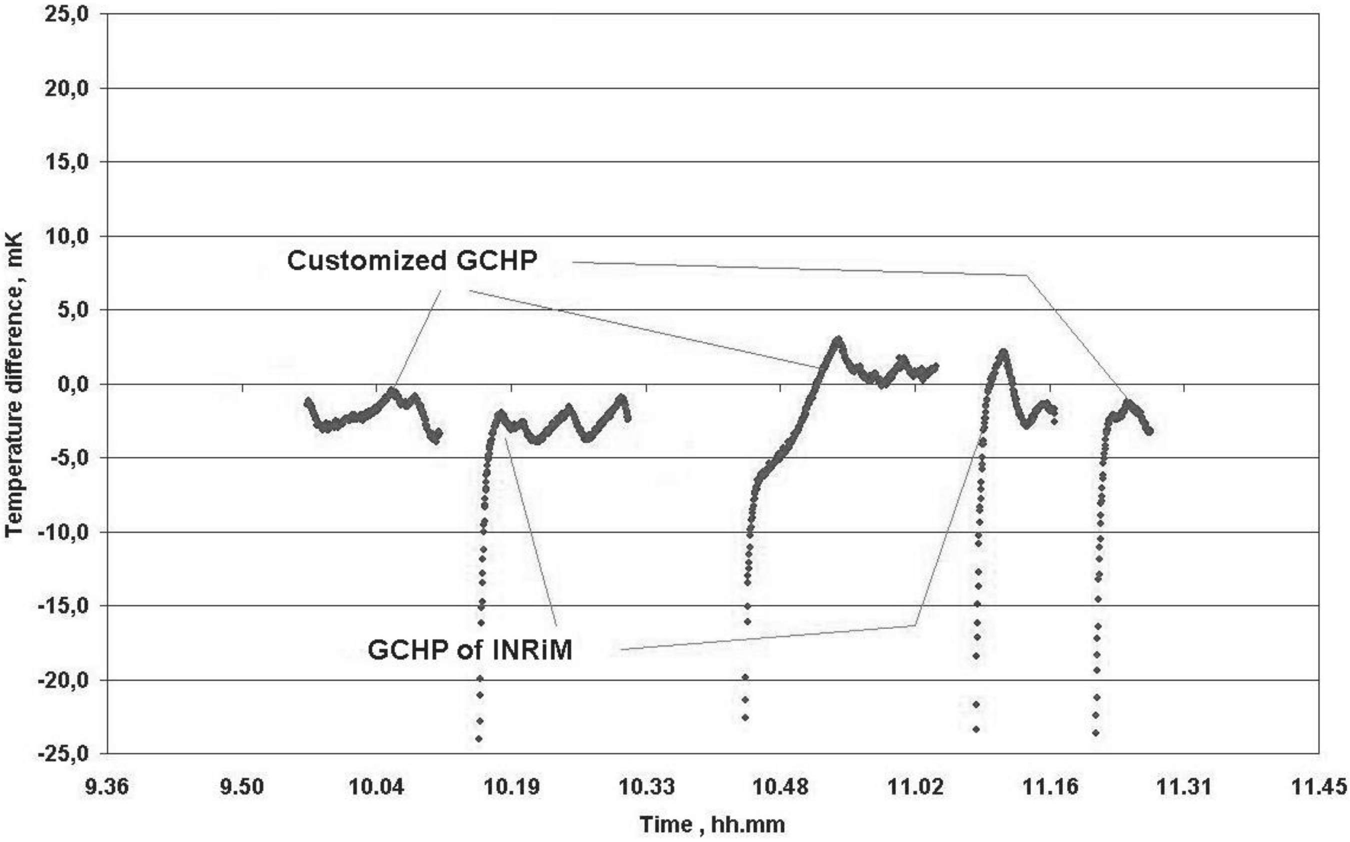


Fig. 6



25,0
20,0
15,0
10,0
5,0
0,0
-5,0
-10,0
-15,0
-20,0
-25,0
Temperature difference , mK
Customized GCHP
GCHP of INRiM
9.36
9.50
10.04
10.19
10.33
10.48
11.02
11.16
11.31
11.45
Time , hh.mm

Fig. 7