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# GAS-CONTROLLED HEAT PIPES IN METROLOGY: MORE THAN 30 YEARS OF TECHNICAL AND SCIENTIFIC PROGRESSES

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## ABSTRACT

Gas-Controlled Heat Pipes (GCHPs) are devices based on generating and maintaining, at millikelvin level, a thermodynamic liquid-vapour equilibrium of a fluid. For this reason, GCHPs have been studied for more than thirty years for research and applications in thermal metrology. Capabilities have been constantly improved and adapted by National Metrology Institutes (NMIs) and accredited laboratories. Activities include study of vapour pressure curves of pure elements and substances, thermometers' non-uniqueness up to 960 °C, calibrations between -20 °C and 900 °C with millikelvin uncertainties, studies of innovative pressure controllers allowing regulation better than $10^{-6}$ from below 1000 Pa up to 400 kPa. GCHPs operating at different temperature ranges have also been connected to a common pressure line in the so-called "Temperature Amplifier" configuration. This review paper presents an almost complete report about the several models of GCHPs, materials and working fluids, techniques adopted in different temperature/pressure ranges. All involved NMIs using GCHP are here included, with detailed bibliography.

**KEY WORDS:** Gas-controlled Heat Pipe, Temperature Metrology, Thermodynamics

## 1. Introduction. The gas-controlled heat pipe.

The Heat Pipe (HP) is a device that transfers heat with a very high thermal conductance, by boiling a fluid at one region and condensing it at another. The concept of HPs has been first introduced by Gaugler [1], in a patent request for a refrigeration system. Grover [2], who for the first time introduced the term “Heat Pipe” described in detail its unusual and remarkable properties. Usually cylindrically shaped, HPs are filled with small quantities of the so-called “working fluids”, depending on the field of temperature and the application needed. In the HP the liquid is transferred back to the boiling area by capillary action through a specific structure along the inner surface.

### 1.1 General overview.

Different kind of capillary structures are adopted: meshes of different textures, ranging from tenths of millimetre to several millimetres are added along the inner walls to force the uniformity of the liquid layer and/or wicks of different sections, shapes and sizes are machined on the internal surfaces. Both the solutions are adopted and in some cases both are present inside the same heat pipe inner walls and thermometer wells (Figure 1). One end of the tube is externally heated, causing the inside liquid to vaporise and the vapour to move to the opposite end of the pipe, which is cooled to condense the vapour back to liquid. The condensed fluid returns back to the heated part of the pipe, due to the capillary forces, thus completing a liquid-to-vapour and vapour-back-to-liquid cycle.

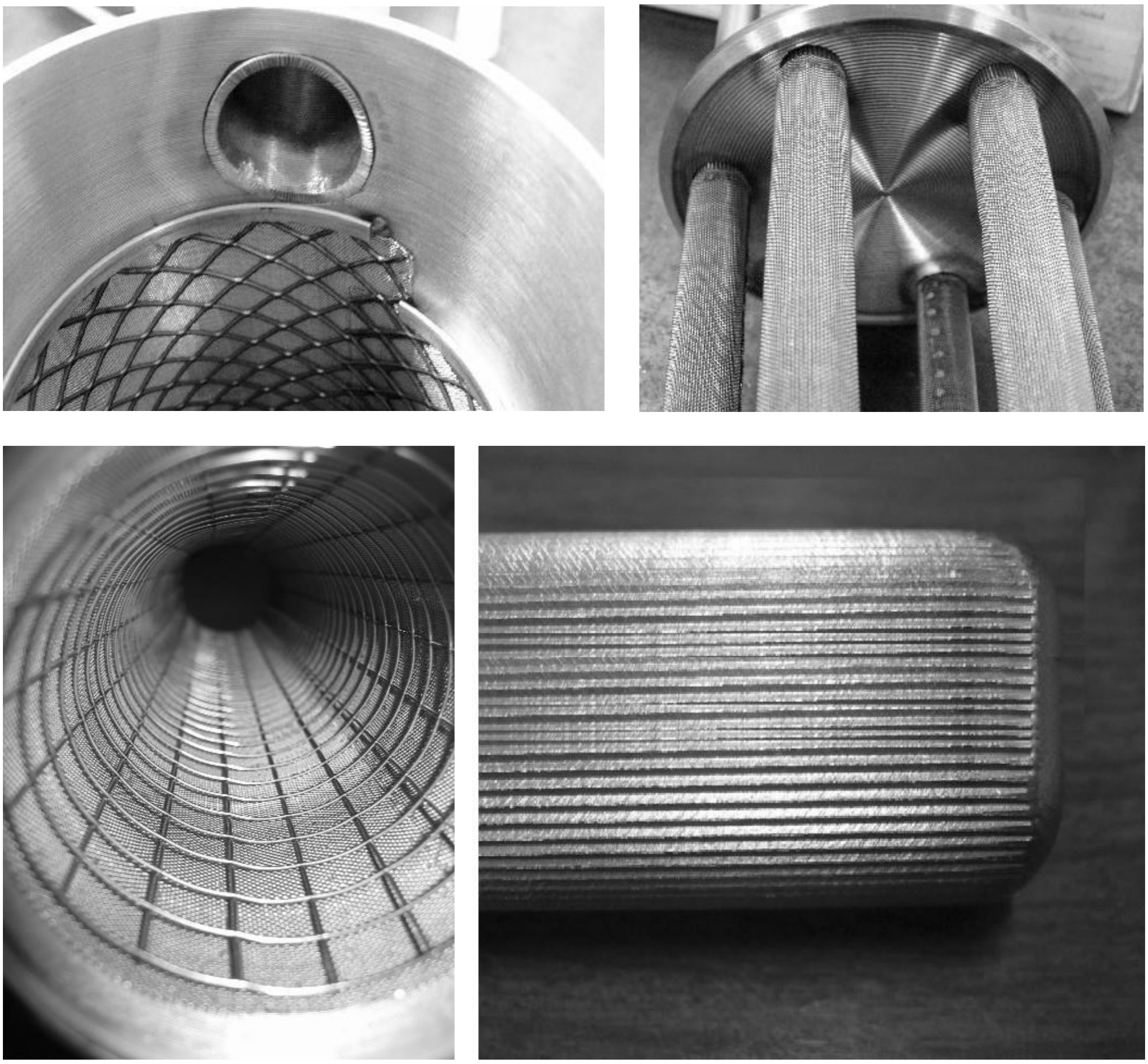


Figure 1. Capillary structure machined (knurls) and added (meshes) inside inner wall and the thermometer wells of a heat pipe. From top left (a) to bottom right (d): (a) view of the lateral chimney connection to the inner volume: an helical structure, is used to keep a mesh in place and uniformly redistribute the fluid coming

back from the cooling zone; (b) six thermometer well attached to the top cover (prior to welding): a mesh for the specific use is fixed around each well; (c) inner wall of a GCHP with capillary structure, mesh and supporting net; (d) close-up of the machined capillary structure around a thermometer well, before applying the further mesh.

The Gas-Controlled Heat-Pipe (GCHP) is a special kind of HP equipped with a gas-control line that enables direct control of the inner pressure (Figure 2). An inert gas such as helium or nitrogen, is used to control the vapour pressure of the working fluid. The gas needs to have a different density from that of the vapour of the working fluid, in order to create an interface between the two. This interface is established at a variable point along the vertical axis of the pipe, depending on some factors such as the heating power, the refrigerating capabilities, the pressure level, the kind of working fluid and control gas used. The inner chamber of the HP is in nearly isothermal conditions, since a stream of vapour, flowing from the heated section to the cooled one, continuously rinses it.

Essentially, it is possible to recognize in a GCHP two interfaces: one between the vapour and the liquid of the working fluid and another between the vapour and the gas. This second interface is placed in the upper part of the HP, closer to the cooled zone. Therefore, the end part of the controlling gas, at position opposed the interface gas/vapour, is almost at room temperature. This makes the gas control process easier, and no particular attention to its temperature is required. The temperature of the interface between vapour and liquid is thermodynamically related to the pressure inside the HP volume. This pressure can be controlled by the gas through the chimney. Therefore, the temperature inside the HP can be controlled and maintained at a desired value by controlling the gas pressure with controllers operating at room temperature on an inert gas.

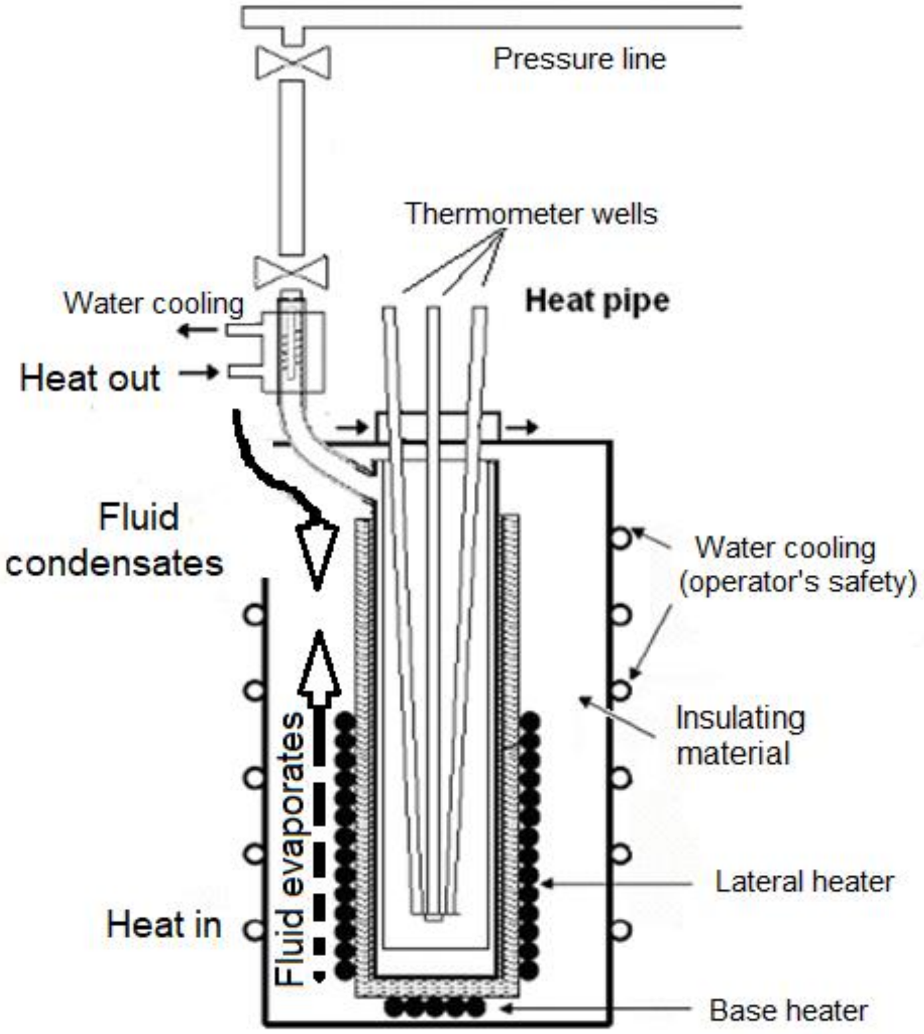


Figure 2. A schematic description of a Gas Controlled Heat-Pipe.

GCHPs used in thermometry are specially developed for the purpose. They are equipped with a lateral chimney used both as the cooling zone and to connect the gas control line. Inside the cylindrical chamber, one or more thermometer wells are placed. The liquid working fluid returns to the heated zone also along knurls machined around the external walls of thermometer wells, in order to keep the measuring zone wet. This ensures a liquid vapour interface all around the thermometer well. Accurate measurements of the liquid vapour phase transition temperature are then obtained by means of Standard Platinum Resistance Thermometers (SPRTs) inserted in the wells. Depending on the planned use, GCHPs have been made with a single central thermometer well (for accurate study of working fluid properties or as temperature generators) up to as many as 12 small size thermometer wells for calibrating multiple sensors, such as thermocouples, at the same time, with respect to a reference thermometer. For calibration purposes, an Inconel® or copper block can also be present, in the vicinity of the expected position of the thermometers' sensing elements, to increase the axial and radial temperature uniformity among the wells. GCHPs and all associated auxiliary equipment, such as the furnace power controller, pressure controller, safety features and cooling lines, are generally quite expensive when considered as complete systems, in comparison with other devices involved in primary or calibration thermometry. Moreover, the intrinsic thermodynamics of the control principle, being quite complicated, require specifically trained staff to properly use this technology and obtain valuable results. On the contrary, when the complete system is working and the operators are accustomed to managing the control principles, the advantages of using GCHPs for research and calibration become immediately appreciable. Better stability and temperature uniformity even at higher temperatures (up to about 1000 °C), quick response times, and shorter times to reach one set point temperature after another are key positive aspects when performing calibrations of large numbers of sensors. Furthermore, for research and development, GCHPs offer a wide breadth of study, for example determining vapour pressure curves, generating thermodynamic relationships, contributing to the improvement of the temperature scale, and characterising thermometers over wide temperature ranges, from below 0 °C up to 1000 °C. While not relevant for calibration purposes, where the actual temperature is given by the reference sensor, in research and development, the purity of the working fluid raises problems in comparability and repeatability. Some of the activities here reported involved working fluids of the highest available purity. For example, a comparison of GCHP capabilities involving potassium from different production lines was performed [3]. It was also discovered, by opening the GCHP, that impurities tend to separate on the top or bottom of the GCHP volume, thus allowing purest working fluid to condense and evaporate close to the thermometers' measuring zone [4].

The capabilities of the gas controlling system are directly responsible for the temperature stability inside the HP and of the extents of the achievable temperature range. GCHPs are equipped with different kinds of pressure controller, ranging from commercial devices, to *ad-hoc* systems based on bellows which change the volume or series of volumes in appropriate ratios, and electro valves. Pressure is normally controlled between around 1 kPa up to 400 kPa. Depending on the temperature range required, different working fluids are therefore adopted, according to their temperature/pressure curve: ammonia, ethanol, water, synthetic materials such as biphenyl, and metals such as mercury, caesium, potassium and sodium. Connecting several heat pipes to a same pressure line, in the configuration of the so-called "temperature amplifier", it allows simultaneous calibration at different temperatures, studies on thermometers and thermocouples and research on thermodynamic properties and vapour pressure curves.

Most GCHPs are manufactured by hand, and very few are off-the-shelf commercial solutions offering standardised equipment. In research, as well as for calibration use, GCHPs are rarely equipped with the same kind of furnaces, the same pressure controllers and the same auxiliary systems (power, safety, cooling lines). Even the inner parts of the heat pipes are made in different ways, according to the expected temperature range, the purpose (research or calibration) and the structure material (stainless steel or Inconel®). To some extent, each system based on a GCHP, its dedicated pressure controller, power lines and controls, cooling and safety circuits represents a unique apparatus. Researchers and technicians who have used GCHPs for calibration or research purposes mainly agree that each GCHP has its own "personality", in terms of response time, stability, reaction to pressure or power changes, uniformity and repeatability.

Small numbers of identical systems have been manufactured and some companies can now offer GCHPs and associated system on catalogue. Different typologies and solutions are described here in more detail.

### 1.2 Dedicated pressure control and performance testing.

The thermodynamic relationship linking temperature and pressure in a fluid during the liquid-vapour phase transition permits an innovative method for very accurate temperature and pressure control. As we have seen, the GCHP is a special kind of heat pipe equipped with a gas line that enables direct control of the inner pressure.

It can be shown from the Clausius-Clapeyron equation and from the ideal gas law:

$$\frac{dT_\mathrm{i}}{T_\mathrm{i}} = \frac{\mathrm{R}T_\mathrm{i}}{ML}\frac{dP_\mathrm{v}}{P_\mathrm{v}} \qquad (1)$$

where $R$ is the universal gas constant, $T_\mathrm{i}$ is the liquid vapour interface temperature, $M$ is the molar mass of the vapour, $L$ is the latent heat of vaporization, and $P_\mathrm{v}$ is the vapour pressure. According to the Pictet Trouton rule, $ML \approx 10\mathrm{R}T_b$, where $T_\mathrm{b}$ is the boiling point of the working fluid; the factor RT/ML can then be approximated as $0.1\mathrm{T}/\mathrm{T}_\mathrm{b}$. Since the GCHPs are generally operated in the pressure region between about 5 kPa and 500 kPa, which corresponds to a temperature variation within ± 20 % of the boiling point, the temperature variation and pressure variation may be linked by the following approximation:

$$\frac{\Delta T}{T} \cong 0.1 \frac{\Delta P_{\mathrm{m}}}{P_{\mathrm{m}}} \qquad (2)$$

A key test was adopted to both check for the correct heat pipe working status and to evaluate time response in terms of pressure/temperature changes (Figure 3). When a pressure change is forced by changing the pressure of the controlling gas, a corresponding temperature change is immediately observed, at a ratio depending on the pressure/temperature relationship for that specific working fluid at that pressure value. The thermodynamic relationship linking pressure to temperature is such that, excluding some mass inertia of the heat pipe body, the temperature change is simultaneous with the pressure step. It is this observed fast temperature response to a pressure change that offers the basis for implementing a thermometer as a pressure sensor.

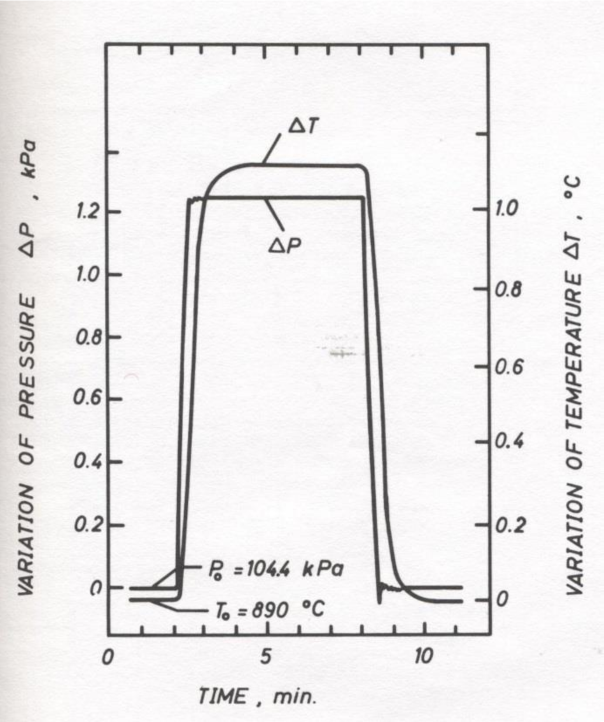


Figure 3a. First evaluations of the transient behaviour of a high temperature gas-controlled furnace [laboratory workbook from Bassani C., 1980].

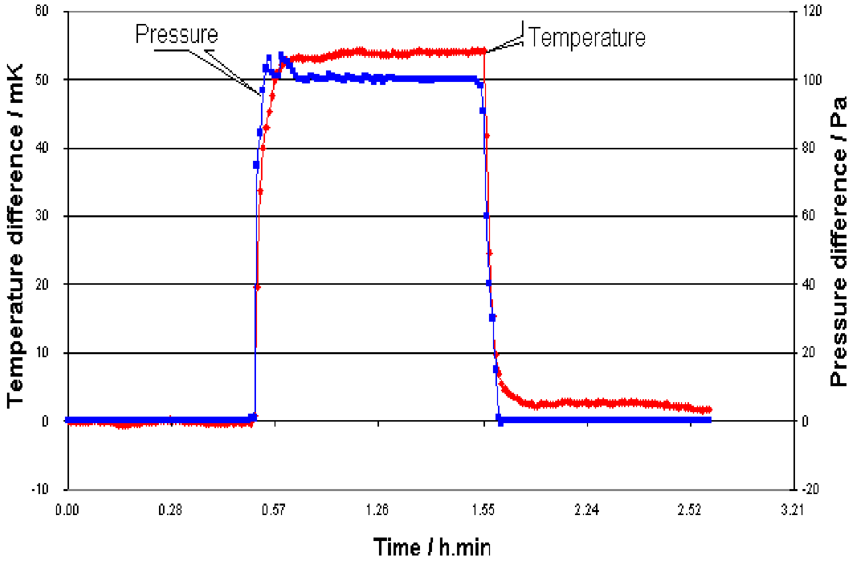


Figure 3b. The same tests are still performed today, to evaluate the response capability and the correct start-up [INRiM technical report 2012].

This physical principle, together with the modern instrumentation and computers fast enough to do the necessary processing in real time allows a new concept in pressure control: the use of the Standard Platinum Resistance Thermometer (SPRT) as a pressure sensor. As we have seen, the accurate measurement of the liquid vapour phase transition temperature is obtained by means of SPRTs inserted in the thermowells. The resistance of the SPRTs is measured with extremely accurate, sensitive resistance bridges (of the order of 1 part in $10^8$). In this case, the set point is no longer a pressure, but a temperature instead, realised by means of a resistance set-point. The quantity under control, then, is the temperature $T$ of the liquid vapour transition of the working fluid inside the GCHP, measured by means of a SPRT that translates the temperature values into resistance values. A simple way to realise control at a desired temperature set-point as a resistance value $R_{sp}$ is to compare it with the measured value $R(T)$ and supply a pressure difference to compensate for it (proportional control). A new dedicated pressure controller, based on volume ratios and computer-driven electro valves, has been studied and developed at INRiM in cooperation with University of Cassino: the system demonstrated capabilities in terms of pressure stability and sensitivity, not so far achievable by any commercially available pressure controller [5]. Once the set point has been defined, the computer, via a resistance bridge, interrogates a SPRT located inside the thermometer well of one GCHP. The software compares the resistance value to the set-point value and configures the electro valves openings and closings appropriately to control the pressure. The smallest pressure variation achievable with the controller is less than 1 part per million (ppm), and the temperature stability can therefore be kept at even better level. This is due to the favourable ratio between relative pressure changes resulting in relative temperature changes of around 0.1 as reported in (2). An example of pressure control using the SPRT to control the temperature/pressure relationship, is reported in Figure 4 and compared with the control capabilities (resolution) of a commercial controller connected at the same time to the same GCHPs.

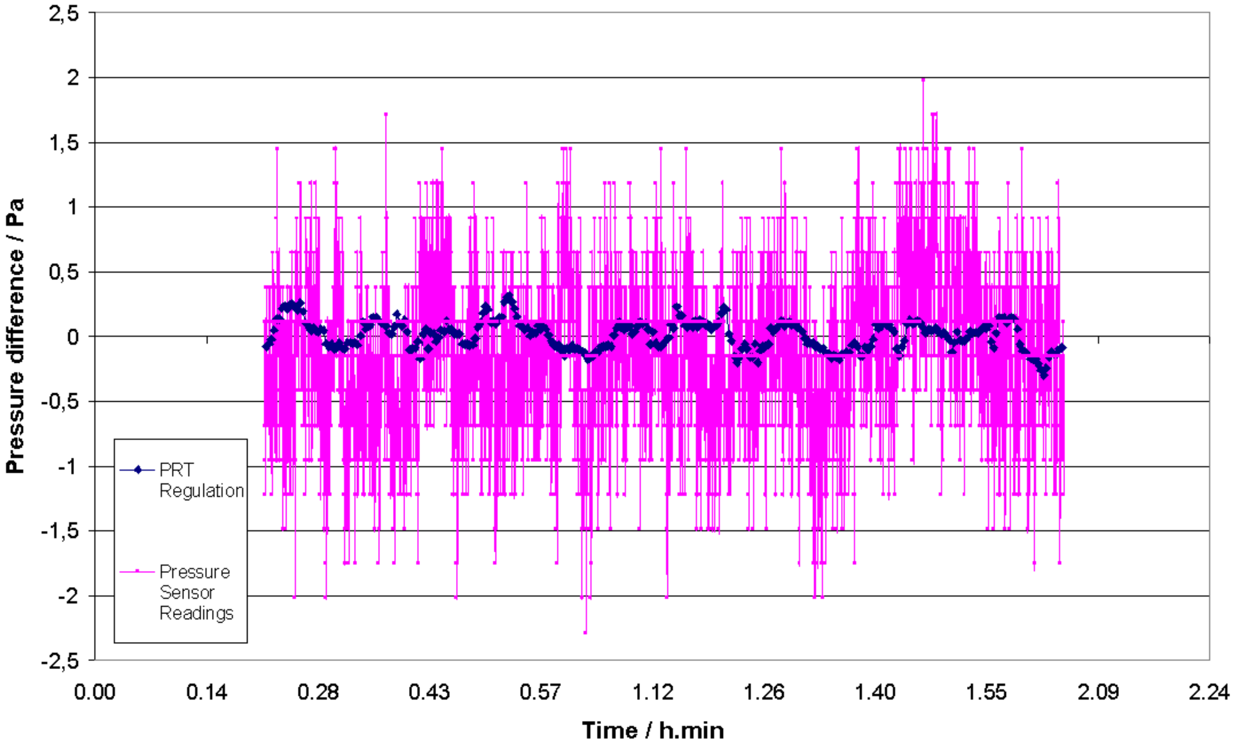


Figure 4. Pressure control achieved with the GCHP dedicated pressure controller developed at INRiM using the PRT as sensor, compared to a top quality commercial pressure controller involving a pressure sensor [28, 30]

## 2. The history of GCHPs in metrology.

### 2.1 The very beginning.

In 1972, at the Euratom Joint Research Centre of Ispra, it was decided to build an improved Couette absolute viscometer to increase knowledge on heat transport fluid properties and processes in nuclear plants. For the housing of this instrument, a thermostat with unusual properties was needed: large volume of 15 l, high temperature homogeneity (0.01 °C), operating temperature up to 140 °C and rapid transition from one temperature to another. These specifications were difficult to meet with the usual liquid bath thermostats. In particular, the transition from one stabilized temperature to another is far from easy with such systems: each time a new isothermal zone has to be found in which to place the viscometer, and these zones become increasingly small with rising temperature. Therefore, the application of the gas controlled heat pipe was considered, on which a fair amount of experience was available at Ispra from work on thermionic space power supplies.

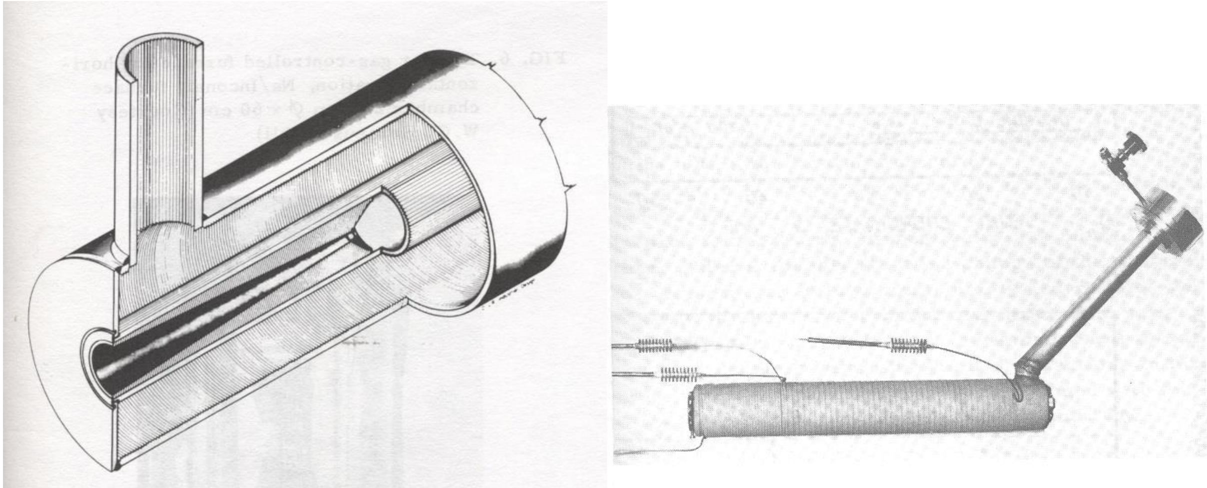


Figure 5. An early published scheme for a GCHP (left) and a GCHP furnace for operation between horizontal and vertical position (Na / Inconel® chamber, 50 cm length, 1.7 cm diameter) [6, 7].

This was the beginning of the development of GCHP furnaces; a scheme and an early picture are shown in Figure 5. The new technology made it easy to meet the requirement for the viscometer [6] in terms of rapidity in changing temperature and reaching a stability of a few millikelvin.
These encouraging initial results then stimulated the extension of the work to higher temperatures where other areas of application of the furnaces were envisaged, e.g. in connection with the effort of improving the International Practical Temperature Scale of 1968 (IPTS-68) between 630 °C and 1064 °C. Caesium and sodium served as working fluids. The investigated temperature ranges were 372 °C to 671 °C and 626 °C to 1068 °C respectively. For the entire investigated temperature range up to 1000 °C the temperature variations were, in general, below the limits of detection of the measuring probe and remained within 10 mK over a length of some 25 cm [7].

In 1982 a fruitful collaboration was launched between the scientists of JRC and those from the Italian Institute of Metrology (at that time the Istituto di Metrologia Gustavo Colonnetti - IMGC) and this made it possible to start to adapt this technology for thermal metrology [8], where accurate, stable, reproducible and spatial homogeneous temperature are the key aspects of any investigation.

### 2.2 INRiM and ISPRA.

Since the 1982 meeting between JRC Ispra and IMGC, the development of new GCHPs, new machining and filling techniques, more accurate vapour pressure investigations started together with the dissemination of the use of such devices to other institutions worldwide. New studies on vapour pressure and to their application for primary temperature metrology where published [9, 10].
When, at the end of the 1990s JRC closed the GCHP production facility, the knowledge was transferred to INRiM where new GCHPs have been studied and manufactured. A dedicated laboratory [11] was then opened in the thermodynamics division and trained personnel have been constantly operating such devices ever since.

The devices are operated as thermometer calibration facilities [12-16], in support of research on the International Temperature Scale of 1990 (ITS-90) [10, 17-21] and for accurate studies on vapour pressure relationships and thermodynamics [22-26].

New GCHPs have been manufactured at INRiM for fundamental resistance thermometry purposes, and are now commercially available [27, 28]. The design of the new heat pipes is an improvement of the design of the heat pipes manufactured some years ago at JRC and takes into account all recent studies carried out at INRiM on these kinds of devices. Inconel® heat pipes are now available for filling with potassium or sodium as working fluids for high temperature measurements. Stainless steel heat pipes have also been manufactured, to be filled with low-temperature working fluids, such as mercury. Six-well and three-well pipes are available, both for higher or lower temperature, on request. A new pressure control system was developed [5]: it has been designed with the express purpose of controlling the pressure inside the heat pipes, by acting on the pressure of the controlling gas.

This controller can use a PRT as sensor, through its reading by an automatic F18 resistance bridge, processed by a computer, and passed on to the controller. This system is able to achieve pressure control even better than the one originally involved and made at ISPRA, but adopts a totally new principle that makes it compact, easier to use and less expensive. This novel pressure controller is based on electro valves separating different capacity volumes and a two connecting lines: a vacuum line and a high pressure line. Appropriate software and real-time processing (now achievable with modern computers) yields performance which surpasses even the capabilities of the one based on bellows. The GCHPs are equipped with dedicated new furnaces, new power-control systems and cooling lines. Two or more GCHPs can be connected to the same pressure control line in order to realise the “Temperature Amplifier” [17, 18] or to obtain continuous temperature ranges for thermometer calibration, from 200 °C to 1000 °C [15, 28].

Several contracts with other institutes and calibration laboratories for providing or improving such devices, dedicated controllers and special software made it possible to reinforce this central role of INRiM. Today INRiM is known as a worldwide reference institute for this kind of technology.

Several Pyrex®-made HPs were manufactured at IMGC to study some of the particular characteristics of those devices. Thanks to the transparency of the Pyrex®, it is possible to have a better idea of what happens inside a HP by examining it visually. The upper cover is easy removable, in order to allow changes inside the chamber. The behaviour of different working fluids and the effect of several kinds of meshes are still under study. In order to study new working fluids and to test wire nets comprised of different meshes, open air controlled HPs made of glass have been realised. Tests carried out on these glass HPs suggested the possibility of using these low cost apparatuses for accurate comparisons of PRTs [13]. The possibility of seeing through the inner part of a heat pipe was also investigated at Ispra, by making small Pyrex® windows on the heat pipe wall. An all-Pyrex® heat pipe extends the visibility to the whole inner volume and surface, allowing an understanding of the behaviour of different capillary structures with respect to different fluids.

In 2005, among other activities, a new GCHP operating with mercury as the working fluid has been designed, manufactured, and completely characterized [28]. This HP is made of stainless steel and is equipped with three thermometer wells. A dedicated furnace has been constructed and specific software algorithms [29, 30] have been implemented for the temperature and pressure control. The complete system demonstrated capabilities at the level of fundamental thermometry requirements: temperature stability could be kept constantly within tenths of millikelvin for any required time duration (Figure 6), temperature uniformity along the thermometer wells was found to be up to 5 mK over 15 to 20 cm (depending on temperature range), and time response was within few minutes for a 10 °C temperature change. Such excellent performance has never been achieved before and is a relevant improvement with respect to the past performance of GCHPs.

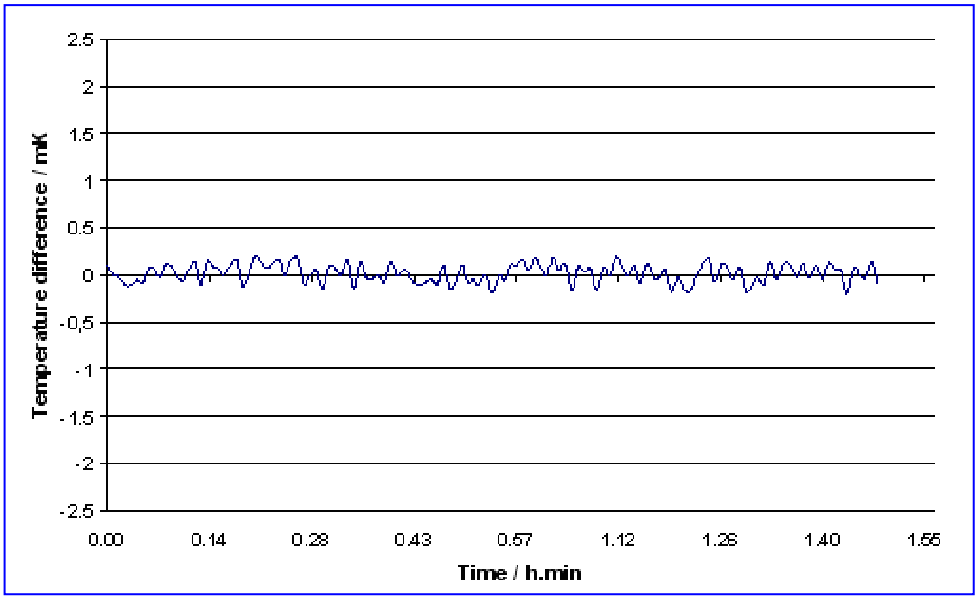


Figure 6. Temperature stability at the level of few tenths of a millikelvin at 370 °C achieved inside the new stainless steel mercury filled GCHP, recorded with a SPRT and an 8-digit resistance bridge equipped with calibrated temperature-controlled standard resistors [26].

This device was then used for multiple purposes. It became the low temperature reference for the new "Temperature Amplifier" (see below); a second one is used as a calibration facility for thermometer calibration by comparison between 220 °C and 450 °C. A third model was used for the most accurate determination of the mercury liquid-vapour curve ever made [26].

| Institute | year | Type of HP and main evolution | Application | Note | Ref. |
|---|---|---|---|---|---|
| JRC | 1972 | Caesium HP (372 °C - 671 °C)<br>Sodium HP (626 °C - 1068 °C) | Improving the IPTS-68 from 630 °C to 1064 °C | Uniformity: within 10 mK at 1000 °C over 25 cm. | [6] |
| JRC | 1978- | Heat Pipe with pressure connection | Study on temperature drift according to pressure change and calorimetry | | [7] |
| JRC<br>IMGC | 1982 | Gas Controlled HP | Study of vapour pressure curves | | [8, 9] |
| NRC | 1988- | Sodium and caesium GCHP | Reference tables for platinum-gold thermocouples | GCHP manufactured by a commercial company | [40, 41, 42, 43] |
| LNE-Cnam | 1994- | Temperature amplifiers | Accurate calibrations of high-temperature SPRTs and thermocouples [31, 32, 34] | Largest number of GCHP connected to a single pressure line. | [31, 32, 34] |
| IMGC | 1996 | Sodium GCHP with six wells<br>Range: 660 °C to 960 °C | Accurate comparison of HTSPRTs<br>Study of the ITS-90 non-uniqueness in the range between Al FP and Ag FP | | [10] |
| NMIJ | 1998 | Water GCHP | Study of the ITS_90 non uniqueness between 65 °C and | First time a comparator block is | [53] |

| | | | | | |
|---|---|---|---|---|---|
| | | | 157 °C [10] | added inside the heat pipe volume | |
| IMGC | 1999 | New GCHPs<br>Inconel HP for high temperature (Na and K)<br>Stainless steel HP for low temperature,<br>Configuration at six or three wells | Thermometer calibration facilities from 200 °C to 1000 °C | | [12,13,14,15,16] |
| NPL | 2000 | Dual sodium-potassium heat pipe with black body cavities | Determination of T-T90 | First link between pyrometry and contact termometry | [59] |
| IMGC | 2001 | Pyrex-made HPs<br>with upper cover removable | Study behaviour of different working fluids and the effect of several kind of mesh<br>Accurate comparison of PRTs | Transparent GHP To study the condensation and capillary effects inside | [13] |
| INRIM | 2007 | New Hg GCHP<br>with dedicated furnace and specific software algorithms for temperature and pressure control. | Low temperature reference for the “Temperature Amplifier”;<br>Calibration thermometers by comparison from 220 °C to 450 °C<br>Accurate determination of the mercury liquid-vapour curve | Uniformity 5 mK over 15-20 cm<br>Stability: < 1 mK | [26, 28, 29, 30] |
| INRIM | 2008 | Temperature Amplifier<br>Hg GCHP (240 °C - 400 °C) and Na GCHP (660 °C - 962 °C) with SPRT with F18 resistance bridge | Studies of the relationship of coupled GCHPs | Best pressure control achieved at the level of $10^{-6}$ | [17] |
| VSL | 2009 | Biphenyl GCHP | Calibration and as low temperature of a prototype of temperature amplifier | Biphenyl used for the first time as replacement for mercury | |
| NMIJ | 2010 | Water GCHP | Evaluation of inhomogeneity of thermocouples | The biggest GCHP ever made | [54] |
| KRISS | 2011 | Pressure controlled pulse heat pipe<br>(PCLHP) | Stability and study on fixed point (tin) | Unique application of a PCLHP in metrology | [46, 47, 48] |
| INRIM | 2015 | Furnace based on K GCHP, with commercial pressure controller (1 kPa to 400 kPa)<br>Range: 450 °C to 900 °C (Ansaldo)<br>GCHP in stainless steel with seven well with biphenyl with commercial pressure controller (Politecnico di Torino) | Use of GCHP outside NMIs: contracts with accredited laboratories | Stability < 5 mK<br>Uniformity < 10 mK<br>1.5 mK/cm on 25 cm. [15]<br>Seven wells GCHPs | |
| LNE-Cetiat | 2016 | Water GCHP | Calibration of thermocouples and industrial thermometers | First heat pipe with removable heat and interchangeable comparator block and thermometer wells. 12 Wells GCHP | [36] |

| | | | | | |
|---|---|---|---|---|---|
| INRiM | 2019 | New sodium GCHP with Inconel block inside | New determination of the ITS-90 non-uniqueness between the Al and Ag points. | | |

Table 1. Chronological summary of the major achievements in GCHP for metrology.

## 3. APPLICATION OF GCHPs.

The capabilities of GCHP in generating stable and uniform temperature volumes allowed their application in various area of the thermal metrology: from calibration of thermometers and thermocouples, to studies on the temperature scale and thermodynamic studies on vapour pressure scales.

### 3.1 Calibration systems.

Calibration by comparison of PRTs and thermocouples requires transfer media capable of providing very good short-term temperature uniformity and temperature stability over a wide temperature range.

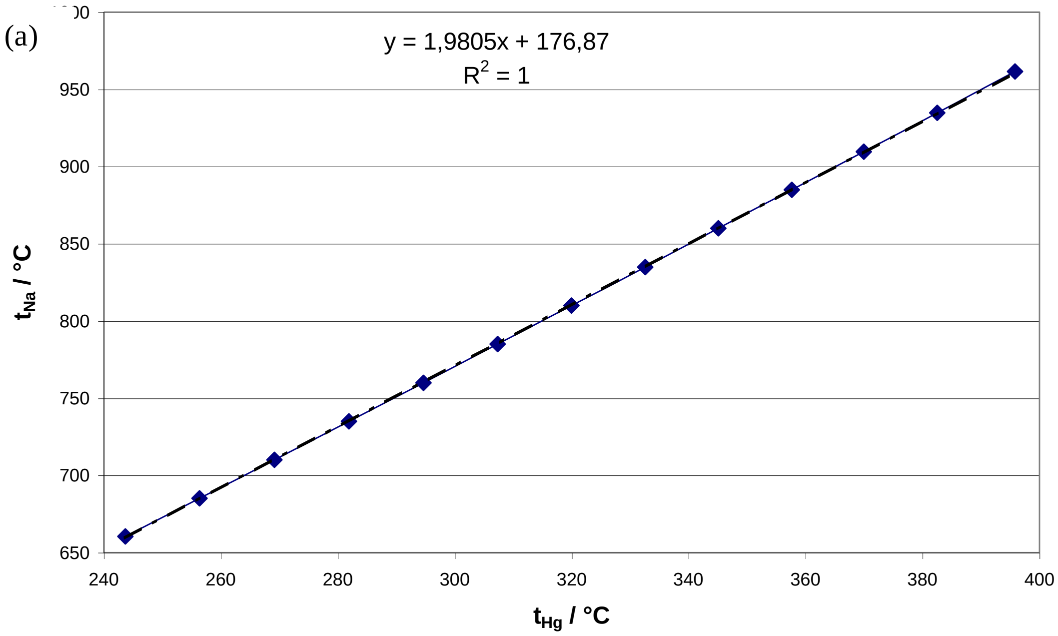
At INRiM, in the laboratory for the comparison calibration of PRTs and thermocouples, a furnace based on a potassium GCHP has been installed (Figure 7). The pressure is controlled by means of a commercial pressure controller operating in the range between 1 kPa and 400 kPa. This GCHP furnace was intended to improve the calibration capability of the laboratory between 450 °C and 950 °C, which was limited at 550 °C by a temperature stability and uniformity of ±0.05 K obtained in salt baths. At higher temperature, furnaces equipped with metal blocks have a temperature stability and uniformity larger than ±0.2 K. The new GCHP furnace has the same design, with six thermometer wells, like the sodium HP previously developed for the comparison of HTSPRTs. In order to extend the operating range to temperatures below 660 °C, potassium is used as working fluid instead of sodium. The pressure is controlled by a commercial pressure controller operating in the range from 1 kPa to 400 kPa. The capabilities of this system were found to be satisfactory for the calibration of resistance thermometers and thermocouples: temperature is stable within 5 mK over hours and constantly within 10 mK for days if needed; uniformity was found to be of the same order among the thermometer wells; the temperature gradient was limited to 1.5 mK/cm in the lower 25 cm of the thermometer wells. The whole system has been renewed over time and it is now in its second generation. Duration tests also showed that after ten years of calibration activity, the old systems maintain a reproducibility very similar to new GCHP, demonstrating almost complete absence of contamination to the working fluid.

Figure 7. GCHP filled with potassium for calibration at INRiM. The system is equipped with a commercial pressure controller [3].

Following the INRiM and LNE-Cnam calibration laboratories, in recent years some calibration laboratories, such as the Ansaldo Energia, the Italian main company on energy plants, or the Politecnico di Torino accredited centres experimented with the use of GCHPs for calibration purposes. Comparison of such devices with those operating at INRiM have been undertaken to guarantee complete operational characterisation and to assess the laboratory procedures and traceably link the vapour/pressure curves to the SI standards. The main advantage of the GCHP, well appreciated by accredited laboratory operators, is the speed of change among temperature points. With similar capabilities to comparator block furnaces and stirred liquid baths, the GCHPs are able to change the temperature and reach the required stability in a significantly shorter time, allowing the user to quickly perform more calibration points. Different models have been studied, designed, manufactured and characterized, in different dimensions, with different numbers and sizes of thermometer wells. The Politecnico of Torino is now operating a seven-well stainless steel GCHP filled with biphenyl with stability of the order of millikelvin and satisfactory uniformity and gradients. Ansaldo is using a potassium filled heat pipe with seven thermometer wells. Both of them use commercial pressure controllers and same power and safety systems. Results have been found satisfactory and now such systems are routinely involved in calibration of thermometers and thermocouples. The chief disadvantage and limitation is presently the complexity of the whole system: power line, pressure control, safety systems and cooling all need care and attention in their design, assembly and use. Specifically trained staff are therefore required to operate calibration procedures involving GCHPs. This is why only few companies are presently offering commercial GCHPs, and those only over a limited temperature range and with a dedicated pressure controller. INRIM is currently undertaking a study on how to reduce the complexity of the system, and how to make available a series of GCHPs for calibration purposes with the best achievable compromise in terms of costs and achievable calibration uncertainties. Aspects such as pressure lines and controllers, dedicated furnaces and safety systems are investigated, with the long-term aim of simplifying the uptake and standardisation of this technology. Besides the key aspect, represented by designing and manufacturing the HP itself, the vision is to adopt, as far as possible, commercial off-the-shelf systems already available, such as furnaces, power controllers, pressure controller and safety systems. The use of the thermometer to drive the actions of the pressure controller is also being studied on the basis of commercial controllers.
Other uses of GCHPs for calibration purposes are also reported in the next chapter 4.

### 3.2 The "Temperature Amplifier".

In 1996, a sodium GCHP with six thermometer wells was used for accurate comparison of high temperature standard platinum resistance thermometers (HTSPRTs), for the study of the ITS-90 non-uniqueness in the range between the aluminium and the silver freezing points [10]. For this use, the temperature inside the HP was directly controlled by means of a High Temperature Standard Platinum Resistance Thermometer (HTSPRT). The drift of the HTSPRT above 660 °C up to the silver point at 960 °C significantly limited the long-term stability of its output. In order to improve the long-term stability and reproducibility, a novel approach was then studied, which consisted of connecting, to the same pressure line of the sodium HP, a HP operating at lower temperatures where thermometers are more stable. The lower temperature HP is then used to maintain a specified temperature achieved by involving a SPRT and a resistance bridge as pressure controller (from the principle described in section 1.2). According to the monotonic relationship between the temperature and pressure of a fluid two during a phase transition process, a controlled stability in temperature corresponds to a pressure stability. This pressure stability generates in turn a temperature stability in the higher temperature heat pipe. Considering the relationships between the mercury and sodium vapour pressure curves, the stability and reproducibility of the low temperature heat pipe is transferred (amplified) to the high temperature GCHP by a factor equal to this relationship (Figure 8). Considering the properties of the SPRTs (reproducibility within millikelvin between recalibrations up to the zinc point), the resolution and sensitivity of the F18 resistance bridge used (one part in $10^8$), and the stability and reproducibility of the mercury heat pipe which are also of the order of 1 mK [28], all mean that the temperature reproducibility of the sodium HP, between 660 °C and 961 °C, surpasses that of the HTSPRTs used for realising the ITS 90, normally of the order of 5 mK to 10 mK at the silver point. This device is generally referred to as the "Temperature Amplifier".



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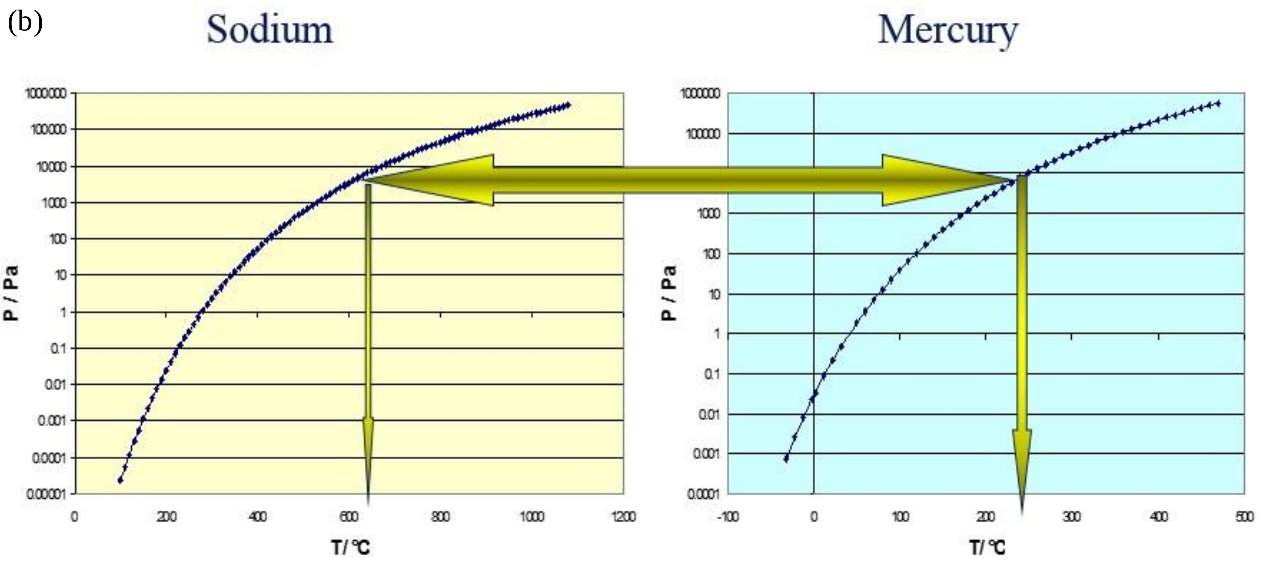

Figure 8. The experimental results for the first prototype of the Temperature Amplifier expressed as a $t_{Na}$ = $t_{Na}$ ($t_{Hg}$) curve between the temperature of the mercury, $t_{Hg}$ and that of sodium $t_{Na}$ at the same pressure (upper graph). Both plots represent the principle of temperature amplification: (a) the first plot shows the linear relationships between the temperatures generated by the two heat pipes; (b) the second plot focuses on the fact that both heat pipes are connected to the same pressure line and realise different temperatures at constant pressure [19].

After having studied properties of candidate working fluids, in 1999 mercury was tested for the lower temperature GCHP. The $t_{Na}(t_{Hg})$ (i.e. the temperature of the sodium HP as a function of the temperature of the mercury HP) relation between the temperature of the mercury, $t_{Hg}$ and that of sodium $t_{Na}$ at the same pressure was determined by simultaneously measuring temperatures inside the two GCHPs, at intervals of 25 K, covering the temperature range in the sodium GCHP between 660 °C and 960 °C (Figure 8). At each point the derivative functions have been measured by varying the pressure $P$ by a small amount (less than 0.1 %), thus obtaining $dt_{Hg}/dP$ and $dt_{Na}/dP$ and then $dt_{Hg}/dt_{Na}$. By integrating the measured derivative curves, the $t_{Na}(t_{Hg})$ curve was re-obtained, within the measurement uncertainties, demonstrating the validity of this relationship, at least between 660 °C and 960 °C, at the level of millikelvins. This data was published by Marcarino and Merlone in 2003. In a more recent study made under in cooperation with NIM (China), a new determination of these relationships was evaluated, also extending the lower and upper pressure ranges. This new investigation involved a complete new system, based on a different pressure controller, new GCHPs filled with sodium from a different batch to those manufactured previously, and all new auxiliary equipment. The study demonstrated, remarkably, that the newly evaluated $t_{Na}(t_{Hg})$ curve agrees with that previously determined within 5 mK over the whole temperature range considered.

In the course of this study on the behaviour of coupled GCHPs, for the first time near-perfect agreement has been obtained between measurements of the immersion characteristics in a mercury GCHP and the Clausius-Clapeyron profile [17]. This result opened up the prospect of using coupled GCHPs to thermodynamically relate two different temperature ranges, with the possibility of redefining one temperature range in terms of another. The temperature in the mercury GCHP can be controlled by controlling the pressure of the inert gas, in this case pure helium, to increase the density difference at the interface with the mercury vapour. The helium pressure is controlled by means of a SPRT inserted in the thermometer well of the mercury GCHP, and using this temperature value as a kind of set point for the pressure controller. In this way, any temperature in the range between 240 °C and 400 °C in the mercury GCHP can be "amplified" in a sodium GCHP connected to the same helium line, to one unique and very reproducible temperature in the range between 660 °C and 962 °C. This method and original approach is now the subject of research cooperation and joint research projects. Studies are also being made to investigate the possibility of substituting mercury in the lower temperature range, with a different fluid to yield the same performance.

As mentioned before, in 2003, Marcarino and Merlone determined the thermodynamic relation between the temperature inside the mercury GCHP ($t_{Hg}$) and the corresponding temperature inside the sodium GCHP ($t_{Na}$) in the Temperature Amplifier [17]. The $t_{Na}(t_{Hg})$ relation was determined by simultaneously measuring temperatures inside the two GCHPs, at intervals of 25 K, covering a temperature range in the sodium GCHP between 660 °C and 960 °C. The $t_{Na}(t_{Hg})$ relation was determined by fitting a third-degree polynomial by means of least squares method over the experimental data. These data were integrated with other available data thanks to the cooperation between INRiM and the Chinese National Institute of Metrology (NIM) [38]. New data, taken with new GCHPs, new heaters, new pressure control systems, and new data acquisition and control software, extends the range of $dt_{Na}/dt_{Hg}$ evaluation at lower pressures and hence lower temperatures. The goal was to compare these two sets of data taken with different devices and assess their compatibility. The experimentally fitted $dt_{Na}/dt_{Hg}$ relation computed from this set of data was then compared with the derivative of the $t_{Na}(t_{Hg})$ relation measured by the previous study (Figure 9), showing differences well within the measurement uncertainties.

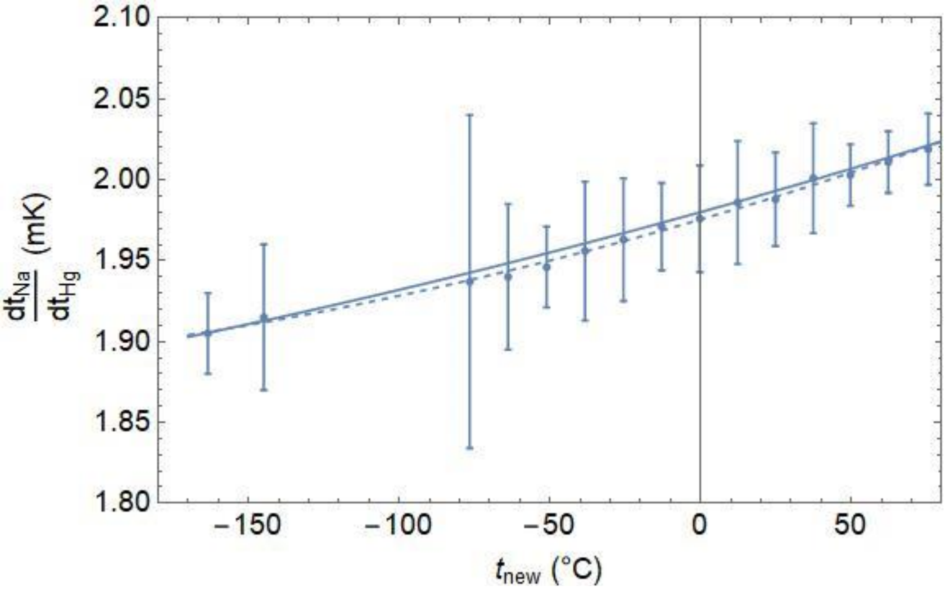

Figure 9. Comparison between the actual fit of the experimentally determined $dt_{Na}/dt_{Hg}$ (dashed line) and the derivative of the experimental $t_{Na}(t_{Hg})$ fit (solid line) showing very good agreement between a series of measurements at millikelvin level (each $dt_{Na}/dt_{Hg}$) with respect to a curve evaluated over 300 °C. $t_{new}$ is a convenient rescaling of the x axis, corresponding to $t_{new} = t_{Hg} - 320$ °C [20].

The integral of the experimentally fitted $dt_{Na}/dt_{Hg}$ relation can be compared with the relation found by the previous study by Marcarino and Merlone, again giving good agreement within few millikelvins (Figure 10).

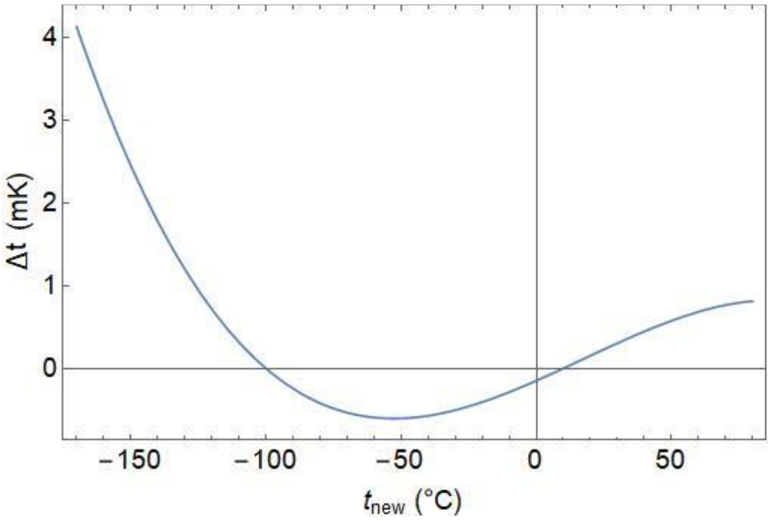

Figure 10. Difference between the $t_{\mathrm{Na}}(t_{\mathrm{Hg}})$ relation by Marcarino and Merlone [17] and the integral of the fit calculated with the whole set of $dt_{\mathrm{Na}}/dt_{\mathrm{Hg}}$ data in 2015. The accordance is within few tens of a mK for a large part of the range (shifted again of -320 °C) [20].

The Temperature Amplifier is a powerful tool to extend the accuracy and the reproducibility of thermometer calibrations. It is possible to transfer the accuracy and stability of the mercury GCHP to the higher temperature sodium one. This could lead to the replacement of the problematic temperature range between aluminium and silver freezing points with any other range, provided the thermodynamic relation between the two ranges are known and characterized with high accuracy; any problems due to the non-uniqueness of high temperature SPRTs can thus be avoided. These results also prove that the relationship of $dt_{\mathrm{Na}}/dt_{\mathrm{Hg}}$ and therefore the $t_{\mathrm{Na}}(t_{\mathrm{Hg}})$ is not dependent on the devices and controlling techniques used to evaluate it.

## 4. A Worldwide Experience.

GCHPs have been used by several national metrology institutes (NMIs) and accreditation laboratories for studies on thermodynamics, thermal metrology and for calibration. Here the most involved NMIs report their experience. This group has been selected to provide key highlights, and is not exhaustive; in particular, it does not include calibration laboratories and other universities using the same devices for other research.

### 4.1 Laboratoire Commun de Métrologie (LNE-Cnam), France.

In the mid-1990s, LNE-Cnam started the development of GCHPs used as temperature amplifiers for accurate calibrations of high-temperature SPRTs and thermocouples [31-33]. The principle is based on the idea that several heat pipes are connected together and to one pressure controller. Each GCHP is filled with a different working fluid so that at a given pressure level, the temperature in each of them is different because the saturation curve (saturation pressure = *f*(saturation temperature) ) is different. Thus, the pressure management of the system is used to shift the temperature of each GCHP, and the temperature range of each GCHP depends only on the substance. To realise the temperature amplifier, one of the GCHPs works over a relatively low temperature range (typically between 40 °C and 150 °C) and houses the reference SPRT, while the other GCHPs work over relatively high temperature ranges (typically between 400 °C and 900 °C) and house the high-temperature SPRTs or thermocouples. In this way, it is possible to carry out a calibration by comparison without exposing the reference SPRT to a high temperature which may lead to drift or damage.

LNE-Cnam carried out studies on a temperature amplifier composed of a HP filled with dodecane (Do), whose temperature was amplified by either sodium-filled (Na) or potassium-filled (K) HPs [34, 35] (Figure11). Figure

12 shows a picture of the temperature amplifier system at LNE-Cnam. Uncertainty budgets for the temperatures realized by each heat pipe were determined and are summarized in Table 2.

Recently, LNE-Cnam developed a new HP filled with pure water, suitable for installation in the temperature amplifier system shown in Figure 12. The water heat pipe is made of copper, and the cylindrical external enclosure has a diameter of 88 mm and height of 600 mm. It is equipped with four 9-mm diameter wells for insertion of thermometers. This design is typical of LNE-Cnam heat pipes.

The water heat pipe was connected to the gas line of the temperature amplifier, where an improved pressure controller made at INRIM [5] was used to ensure the regulation of the pressure with optimal stability and reproducibility (Figure 13). The system was used to “amplify” the temperature generated by the water heat pipe, working between 80 °C and 120 °C, with a sodium heat pipe whose corresponding temperatures ranged between 799 °C and 970 °C. Pressures were between 40 kPa and 200 kPa.

Figure 11. Typical design of a GCHP developed at LNE-Cnam [34, 35].

Figure 12. Temperature amplifier at LNE-Cnam, composed of six heat pipes filled with dodecane, water, sodium, potassium and caesium on a common gas line, connected to a pressure control system. The pressure control system has been replaced in 2006 by an improved controller developed at INRIM that involves a thermometer as pressure controller [32, 33].

Table 2: Uncertainties on temperatures realized with dodecane, potassium and sodium heat pipes at LNE-Cnam.

| **Uncertainty component** | **Do<br>190 °C – 250 °C** | **K<br>600 °C – 840 °C** | **Na<br>799 °C – 965 °C** |
|---|---|---|---|
| Pressure measurements | 1.9 mK to 4.4 mK | 7 mK to 3 mK | 8 mK to 4 mK |
| Calibration and reproducibility of (HT)SPRT | 2 mK | 1.7 mK to 4 mK | 4 mK to 7 mK |
| Temperature stability and uniformity of working volume | 7.3 mK | 8 mK | 9 mK |
| Combined standard uncertainty | 8 mK | 10 mK to 11 mK | 12 mK to 12.7 mK |

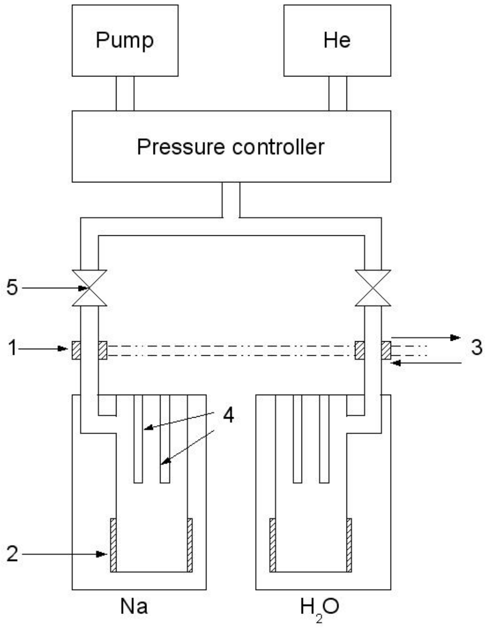

Figure 13. Scheme of the temperature amplifier with water and sodium heat pipes. 1: water cooled section, 2: heating resistor, 3: water, 4: thermometer wells, 5: valve [31].

The system was characterized by using the pressure controller provided by INRIM. Pressures were applied on the gas line by the pressure controller and the corresponding generated temperatures were measured with calibrated thermometers on both heat pipes. In this first configuration, the feedback signal for the pressure control was generated by a pressure sensor included in the controller. Hence, the stability of the pressure in the line was mainly related to the sensitivity of the pressure sensor. The work was carried out in close cooperation with INRIM. The temperature amplification relationship between water and sodium temperatures was determined as follows:

$$T_{\mathrm{Na,amplified}} = A \cdot \left[ \frac{T_{\mathrm{H_2O}}}{T_0} - 1 \right]^2 + B \cdot \left[ \frac{T_{\mathrm{H_2O}}}{T_0} - 1 \right] + C$$

where:

$A = (16.46 \pm 0.16) \times 10^{-4}\ °C^{-1}$
$B = 396.604 \pm 0.015$
$C = 883.082 \pm 0.004\ °C$
$T_0 = 100\ °C$

Additional measurements were then carried out to validate the determined temperature amplification relation. In this second configuration, the feedback signal for the pressure controller was the temperature measurement carried out with the low temperature thermometer in the water HP. This improved the stability of the pressure control, because the sensitivity of an SPRT is higher than that of the pressure sensor installed in the controller. In consequence, temperatures generated in the high temperature heat pipe showed an improved stability as well. As for the determination of the temperature amplification relationship, calibrated thermometers were used again in both the heat pipes. In this way, it was possible to calculate the difference between $T_{\mathrm{Na}}$ determined by amplification and $T_{\mathrm{Na}}$ determined according to the ITS-90 (i.e. the temperature measured in the sodium heat pipe as resulting from the thermometer calibration). The difference $T_{\mathrm{Na}}$, ITS 90 – $T_{\mathrm{Na}}$, amplified was less than 35 mK at all the measured temperatures, and the associated uncertainties were less than 50 mK, as reported in Table 3.

Table 3: Differences and associated uncertainties between the calibration temperatures and the temperatures determined with the temperature amplification relation.

| Temperature in Na heat pipe °C | $T_{\mathrm{Na,\ ITS\text{-}90}} - T_{\mathrm{Na,\ amplified}}$ °C | Expanded uncertainty $U(T_{\mathrm{Na,\ ITS\text{-}90}} - T_{\mathrm{Na,\ amplified}})$ °C |
|---|---|---|
| 799 | -0.029 | 0.034 |
| 818 | +0.022 | 0.034 |
| 840 | -0.006 | 0.034 |
| 902 | -0.005 | 0.035 |
| 922 | +0.005 | 0.035 |
| 944 | -0.033 | 0.035 |

### 4.2 Centre Technique des Industries Aérauliques et Thermiques (LNE-CETIAT), France.

CETIAT is a calibration laboratory accredited by COFRAC. The temperature generators are stirred calibration baths (from -80 °C up to + 215 °C), dry blocks and furnaces (from -90 °C up to +1050 °C) and thermostatic chamber (from -30 °C up to 160 °C). The best calibration uncertainty is achieved in a calibration bath; for PRTs it is 0.03 °C. Nevertheless, the daily calibrations lead to an uncertainty about 0.06 °C for industrial Resistance Temperature Device (RTD) sensors. CETIAT is working on the implementation of a GCHP temperature generator to replace the thermometric baths in order to increase productivity and in order to improve temperature stability in the working volume of the generator. For this purpose, the new system must have better thermal performance and produce the same temperature range as the stirred baths.

In 2016, CETIAT characterized a water GCHP, developed according to the INRiM design, for industrial applications to cover the temperature range from 30 °C up to 150 °C [36]. The results were in good agreement with the expectations and water GCHP started to be used for calibration work. However, there is a need to extend the temperature range to lower temperatures. The top cover can be removed and replaced with different configurations and sizes of thermometer wells. Comparator blocks can be added or removed at the bottom of thermometer wells to respectively improve temperature uniformity or speed. To operate below room temperature, this heat pipe is kept in a liquid bath. The chimney (copper piping in the top right of Figure 14) is further cooled by a circulating bath.

In 2018, CETIAT characterized a new ethanol HP. This is effectively the same heat pipe as before but with the water replaced by ethanol. This system allows the generation of temperatures between -40 °C and 30 °C in the low temperature enclosure.

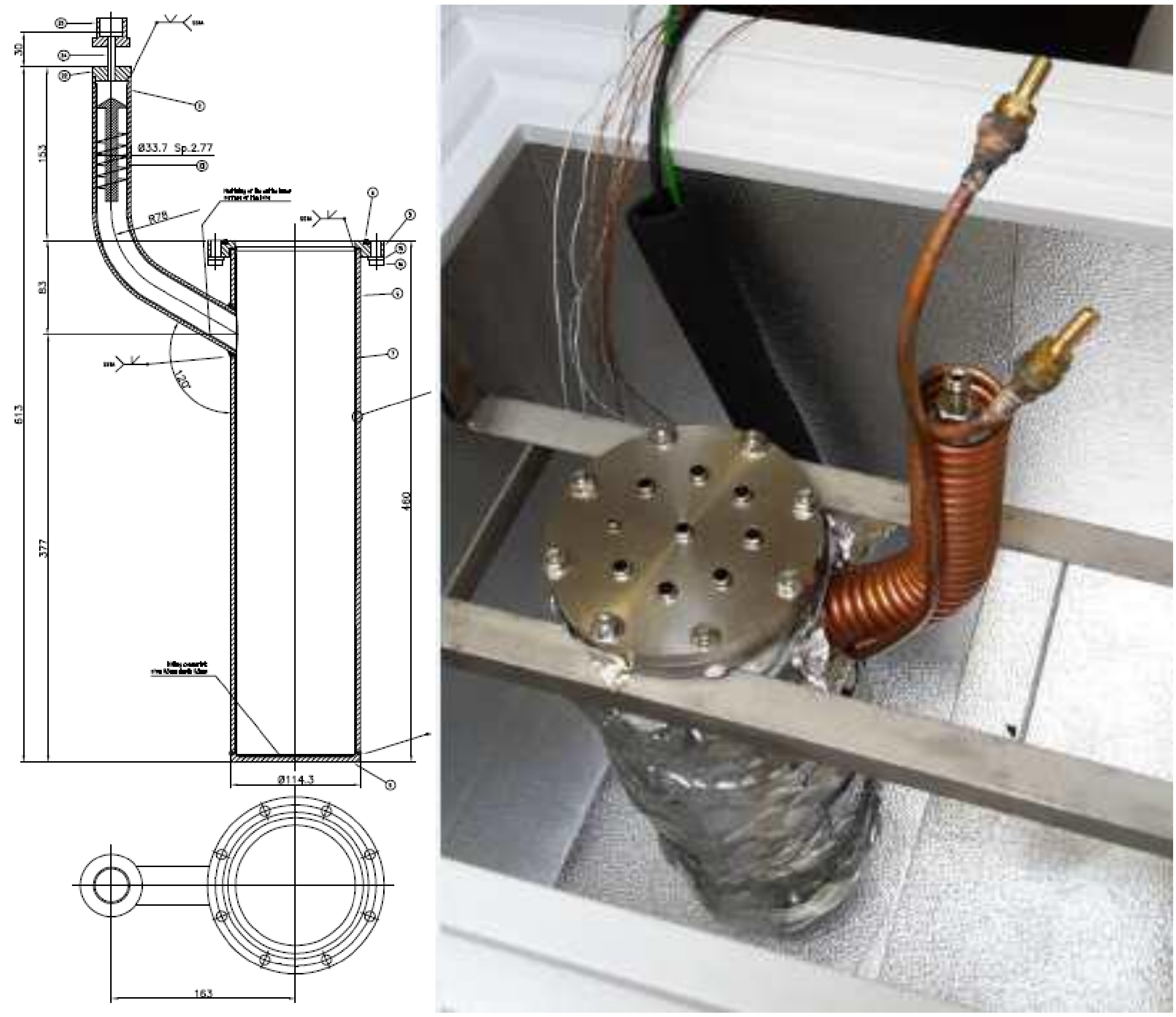

Figure 14. Scheme (left) and picture (right) of the GCHP used at LNE-CETIAT [36].

When using water as the working fluid, this heat pipe showed a temperature homogeneity of no more than 4 mK among all the well at temperatures between 30 °C and 90 °C, and a temperature stability of 5 mK maximum in the same temperature range. When using ethanol, the operating range was lowered to -20 °C to 20 °C with similar homogeneity among the wells and stability of about 7 mK, although with worse vertical homogeneity, due to the lighter working fluid and some minor heat conduction. To make available both ranges, in September 2019 CETIAT purchased a new GCHP from the same company with similar design and under technical supervision of INRiM. It will be tested with other working fluids to reach lower temperatures or with biphenyl, depending on the requests from the laboratory.

## 4.3 National Physical Laboratory (NPL), United Kingdom.

A further use of heat pipes in practical thermometry concerns the evaluation of the thermoelectric uniformity of thermocouples; insertion of thermocouples into a heat pipe enables a direct measure of the combined Seebeck coefficient of the thermocouple as a function of position along the sensor. This in turn enables users to characterise the measurement uncertainty associated with thermocouple use, as well as to gain insight into mechanisms underlying calibration drift during use. A dual GCHP system, consisting of one potassium and one sodium GCHP, is currently also being used at NPL in order to carry out measurements of the difference between the ITS-90 and thermodynamic temperature over the range from 420 °C to 1000 °C. Each heat pipe incorporates a blackbody cavity at one end, so that thermodynamic temperature can be measured using an absolutely calibrated radiation thermometer, and thermometer wells at the other end, to accommodate contact thermometers (SPRTS and Au/ Pt thermocouples) for measuring ITS-90. [59]

For measuring thermoelectric inhomogeneity, it is necessary for the isothermal enclosure to have good temperature uniformity and stability. This was determined for the apparatus at NPL at 600 °C, over the whole length using an industrial PRT [37]. A linear slope spanning 3 °C was observed from the entrance to the exit of the furnace, which may be due to radiative heat transfer ('light piping') within the quartz tube. The stability of the furnace over a period of about 4 h was within a few millikelvin. The effect of the temperature gradient of 3 °C is negligible because it results in a maximum absolute error in scanning a 700 mm long thermocouple amounting to the magnitude of the non-uniformity divided by the scanning temperature (in units of kelvin), or 1.5 K / 873 K, which amounts to 0.17 %, quite acceptable for homogeneity measurement purposes. The value of 1.5 K is used as the tip does not extend beyond half the useable length. This non-uniformity is expected to increase as the temperature increases, but the fractional error should remain approximately constant.

## 4.4 National Institute of Metrology (NIM), China.

In August 2006, a scientific cooperation agreement between INRiM and NIM was signed [38]. Under this cooperation, INRiM manufactured, filled, characterized and delivered to NIM two gas-controlled HPs, one filled with mercury and one with sodium to be connected together in the Temperature Amplifier configuration (Figure 15). During the period from 2009 to 2011, a further gas-controlled sodium HP system was successfully developed at NIM [39]. Also, investigations into the thermal characteristics of the NIM gas controlled sodium HP were carried out. The temperature stability over 5 h was better than ±0.25 mK while controlling the pressure at 111250 Pa. The temperature uniformity within a distance of 14 cm from the bottom of the thermometer well was within 0.3 mK. While keeping the pressure stable at the same value, 17 temperature determinations were performed over 14 days, yielding a temperature reproducibility of 1.27 mK. Additionally, the NIM gas controlled sodium HP was compared with the sodium HP produced by INRiM.

Figure 15. Photo of the NIM Sodium HP [39].

The temperature in the INRiM sodium HP operating at 111 250 Pa was determined, yielding a difference of 21 mK with respect to the NIM GCHP. This difference was attributed to sodium impurities, pressure controller capabilities and reproducibility, and instabilities of HTSPRTs. Further investigations will be carried out extending the pressure/temperature range and connecting both GCHPs to the same pressure line.

### 4.5 National Research Council of Canada (NRC).

The NRC first sodium gas controlled heat pipe was purchased from Dynatherm in the 1980s. A few more sodium and caesium gas controlled heat pipes of various design (one end closed, cooling jacket on the chimney, location and number of the thermometer wells, etc.) were acquired in the 1990s. During that time, various pressure control and power distribution schemes were investigated. Finally, a bellows and needle valve system was developed using a calibrated standards-level deadweight gauge in the pressure control feedback loop to give accurate and stable absolute pressure measurements. This technique delivered outstanding temperature stability and a thermowell immersion gradient of +/- 1 mK over 25 cm was realized. The system remained in operation until 1995 and was used for measuring the vapour pressure curve of Na and Cs and for producing the reference tables for platinum-gold thermocouples [40-44].

In 2017, the original caesium- and sodium-filled gas controlled heat pipes used in the work cited above were recommissioned with a number of changes to improve their function as a comparison apparatus, including a new gas handling system (Figure 16), a new pressure sensor with a quartz crystal resonator for robust stable measurement of pressure, DC power supplies and a dedicated furnace for sodium gas controlled heat pipe which can be operated both in horizontal and vertical orientation. It was demonstrated that the new gas handling system is capable of controlling the pressure in the Cs-PCHP within ±4 ppm of the set point for days, if necessary, as illustrated in Figure 16.

Additional tests to characterize the gas controlled heat pipe performance as a comparison apparatus are currently under way. In the meantime, its exceptional temperature stability – it is possible to maintain the temperature for days within a few mK in the 370 °C – 965 °C temperature range – was used to successfully evaluate the drifts in new types of thermometers to a level better than 2 mK/h.

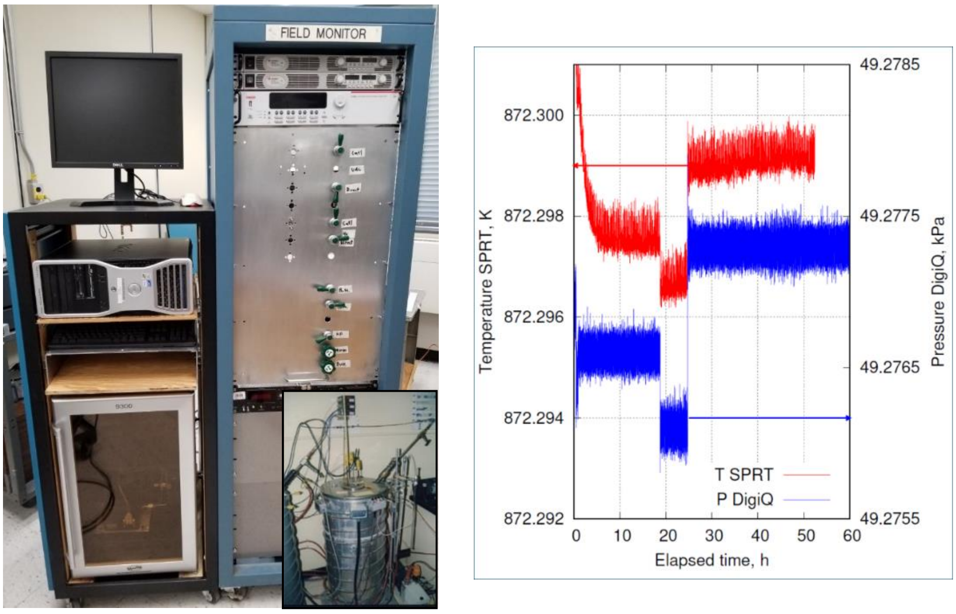

Figure 16. Left: new NRC gas handling system for using GCHP as a calibration by comparison apparatus. Right: long-term stability of the legacy Cs GCHP (inset) when used with the new gas handling system [40-44].

### 4.6 Van Swinden Laboratory (VSL), Netherlands.

VSL use two GCHPs as a temperature amplifier. While the high temperature is the “usual” sodium-filled GCHP, for the low range VSL adopted biphenyl as the working fluid, instead of mercury, to overcome safety regulations on the use of mercury. The helium pressure is regulated by means of a commercially available pressure controller. This allows any temperature in the range between 158 °C and 290 °C to be "amplified" in a sodium HP connected to the same helium line, to one, unique and very reproducible temperature in the range between 660 °C and 962 °C. The biphenyl and sodium HP at VSL have been characterised for temperature stability, uniformity along the wells, and response times over the whole operating range.

The HP setup consists of two temperature-controlled furnaces in which the heat-pipes are placed. The temperature of the furnace is controlled by a Eurotherm controller. The sodium HP contains six stainless steel thermometer wells containing a Carborundum® insert. The Carborundum® inner tubes protect the (S)PRTs from contamination originating from the stainless steel thermometer wells. The biphenyl heat-pipe contains six stainless steel thermometer wells (no inserts) and is filled with 330 g of biphenyl. The power required to maintain the liquid/vapour phase transition inside the GCHP is provided by a one-zone furnace with side heaters but without bottom heating.

The pressure control system for the GCHPs is equipped with a commercially available PPC2+ pressure controller from DH Instruments provided with a high-pressure line to increase the pressure, and with a low-pressure line to decrease the pressure. The high-pressure line of the controller is connected through a pressure regulator to a pressurised helium cylinder, while the low-pressure line is connected to a vacuum pump. The controlled-pressure line is connected to the GCHP through a 25 l buffer volume that is thermally insulated. This buffer attenuates any pressure fluctuations that may be due to perturbations. Temperature stability within a few millikelvins is achieved at intermediate temperatures between 180 °C and 250 °C, where the system exhibits the best performance due to the pressure control capabilities decreasing at the upper and lower range limits.

Two separate cooling lines are in place. The first line provides the necessary cooling to the chimney of the GCHP, in order to keep it working properly; it represents the fundamental cooling that causes the vapour to condense back to the liquid state and return to the bottom of the GCHP. The second water-cooling line is used to cool the outside of the furnace to protect operators. 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.

Using the commercial pressure controller, it is possible to obtain a reasonable stability at any pressure in the range studied. The PPC2+ controller has a specified stability of ±0.003 % of reading which is equivalent to 1 mK at 100 kPa (254 °C) for biphenyl. The measured temperature stability was found to be within 10 mK with a standard deviation of 2 mK. The gradients in the uniform zone between 5 and 20 cm from the bottom were found to always be smaller than 2 mK/cm down to less than 1 mK/cm in some thermowells.

Since the biphenyl heat pipe can operate in a similar temperature range to the mercury heat pipe ([26]) and is a candidate to replace mercury, now widely restricted due to its toxicity, it is worth comparing and commenting on the performance of the two systems. The temperature uniformity of this the biphenyl heat pipe is better by a factor of two than the one evaluated in the mercury heat pipe, while the temperature stability is worse (2 mK to 10 mK compared with a few tenths in the mercury one). The better uniformity is a worthwhile feature, while the worse stability comes from the compromise of having a system that is easier to use, which is driven by a commercial pressure controller, instead of one with the thermometer, bridge and dedicated pressure system. The temperature stability in the GCHP is in fact directly linked to the achieved pressure stability which, in the case of the mercury GCHP was pushed at highest levels, involving complex and more expensive equipment and control processes. Overall, these characteristics are satisfactory for the use of this system for commercial calibrations.

### 4.6 Physikalisch-Technische Bundesanstalt (PTB), Germany.

The Physikalisch-Technische Bundesanstalt (PTB) is the German national metrology institute and the highest national technical authority for metrology. The “Temperature” department is responsible for the realization of the ITS-90 from 0.65 K to 962 °C and its dissemination by using contact thermometers up to 1800 °C. The PTB has used HPs as isothermal furnace liners for more than three decades to realize the fixed-points of ITS-90. These HP furnaces cover the range from the freezing point of indium (≈ 157 °C) to the freezing point of copper (≈ 1085 °C) with different working fluids. Recently a leading manufacturer of HPs and PTB have jointly developed a measurement setup, called the Very Stable Temperature Comparator (VSTC), which is based on a gas controlled heat pipe [45] (Figure 17). Such systems combine the high thermal homogeneity of heat pipes with excellent temperature stability, achieved through the control of the working fluid pressure [20, 34, 45]. Due to the approximately exponential increase of the saturation vapour pressure with the temperature, a relative pressure change results in a relative temperature change approximately ten times smaller. This allows an extremely precise temperature adjustment.

The VSTC is based on a sodium HP made of a nickel-based alloy, which enables operation between 600 °C and 1000 °C. First test runs of the PTB system show a standard deviation of 1.4 mK over 30 min at 1000 °C. This allows very accurate comparisons of up to five thermometers between the aluminium (660.323 °C) and silver (961.78 °C) fixed-points of the ITS-90, which is of great interest for the thermometry community.

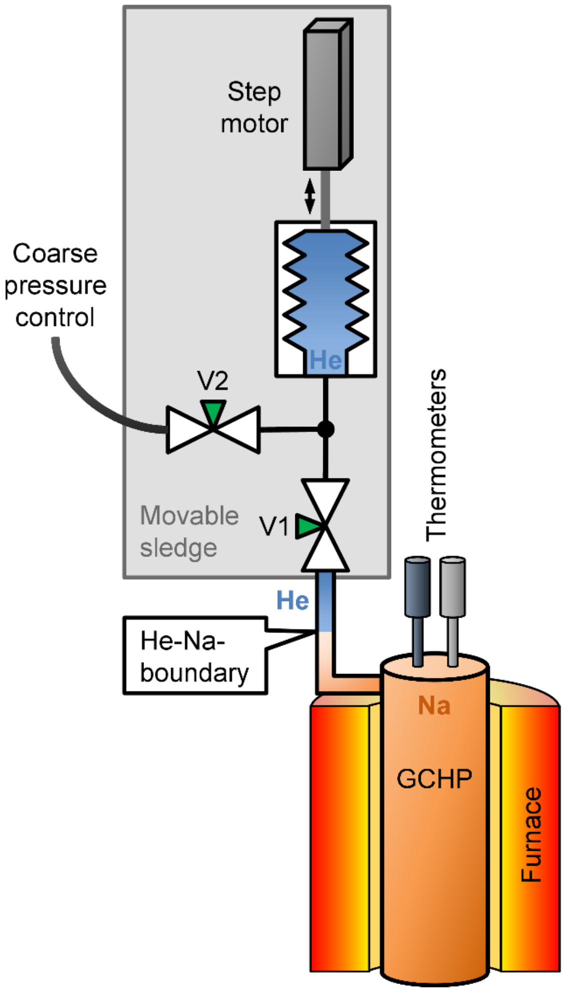

Figure 17. The GCHP system at PTB with closed pressure control system via step motor and bellow [45].

### 4.7 Korea Research Institute of Standards and Science (KRISS).

At KRISS (Korea Research Institute of Standards and Science), a novel temperature control technique, which was based on a different type of HP (i.e. the loop heat pipe), was developed [46-48]. Loop heat pipes (LHPs) are highly promising passive two-phase heat transfer devices, and differently from the conventional heat pipes, they have unique structural characteristics of a physically separated evaporator from an additional two-phase working fluid reservoir (i.e. the compensation chamber). As these two components are thermo-hydraulically linked, the temperature of the vapour phase working fluid generated in the evaporator is determined where the thermal and hydraulic balances are attained between these two components. In addition, because of the physical separation of the evaporator from the compensation chamber, which acts as a non-condensable gas accumulator, the LHPs remain stable even with a large amount of the control gas insertion or withdrawal into or from the compensation chamber. As a result of this feature, fast and stable temperature control is attainable by means of the pressure change of the control gas in the compensation chamber (i.e. the hydraulic temperature control technique).

In the recently developed pressure-controlled LHP (PCLHP) designed for the hydraulic method of temperature control, a cylindrical space for a fixed-point cell was formed in a high-speed vapour flow region (i.e. the isothermal region), and its temperature was controlled by changing the pressure of an immiscible control gas (i.e. He) in the compensation chamber [47, 48]. Figures 18 (a) and (b) show a schematic of the PCLHP and the typical result of the hydraulic temperature control, respectively. In figure 18 (b), the response of the isothermal region temperature ($t_{\mathrm{I.R.\ reading}}$) to the pressure changes of the control gas ($p_{\mathrm{control\ gas}}$) was shown together with the predicted isothermal region temperature ($t_{\mathrm{I.R.\ prediction}}$) which was calculated based on the theoretical relation (derived from the Clausius-Clapeyron approximation). As shown in the figure, the isothermal region temperature was instantaneously and stably controlled by the pressure change of the control gas, and the measured isothermal region temperature was satisfactorily predicted by the theoretical relation.

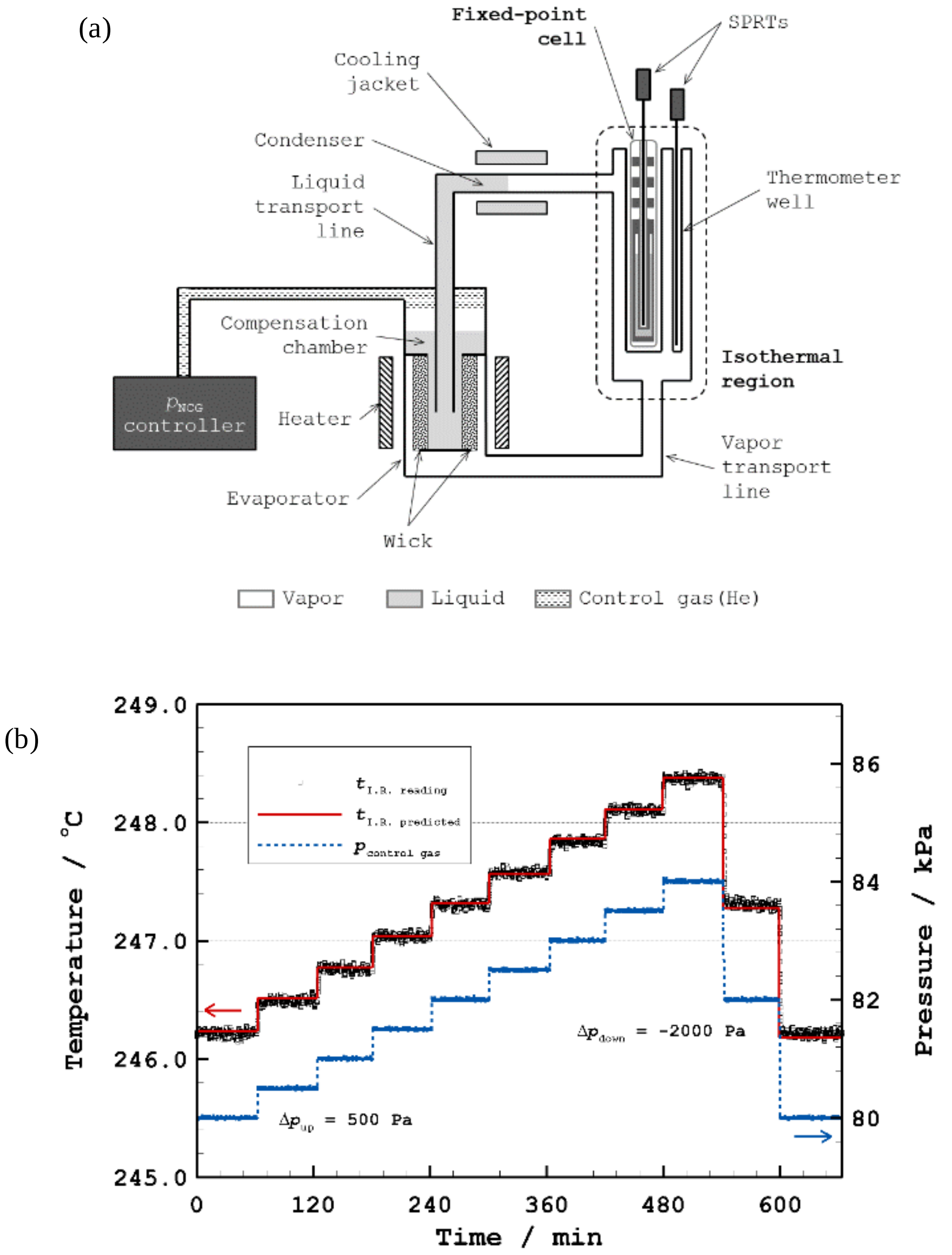

Figure 18. (a) Schematic of the pressure-controlled loop heat pipe [52] (b) Typical examples of the hydraulic temperature control [47].

As a metrological application to the realization of the ITS-90 fixed points, the LHP-based temperature control technique was used to realize the freezing temperature of tin (Sn FP) by means of the inside nucleation technique, where the supercooling and the nucleation was achieved by controlling the isothermal region temperature very fast and stably without extracting the cell out of the isothermal region; as much as 0.37 mK higher freezing temperature ($U$ = 0.19 mK ($k$ = 2)) was measured compared to the conventional outside-nucleated Sn FP [46]. Recently, the LHP-based temperature control method was employed to generate periodic square wave-type temperature steps, which were required to determine the liquidus temperature of the fixed-point metal using the heat pulse-based melting. The liquidus temperature of the fixed-point metal was determined by extrapolating the melting temperatures to the melted fraction of unity. Figures 19 (a) and (b) show the generated periodic temperature steps and the melting temperature variations of the tin samples having segregated and uniform impurity distributions, respectively. The extrapolated melting temperature of the segregated sample was 0.95 mK higher than the outside-nucleated Sn FP ($U$ = 0.15 mK ($k$ = 2)), and it was supposed to be a closer approximation to the liquidus temperature of tin [50]; the effectiveness of the proposed method was further corroborated by comparing the melting temperatures at two different pressures (i.e. 101 325 Pa and vapour pressure of tin) on the basis of the pressure effect given by the ITS-90 [51].

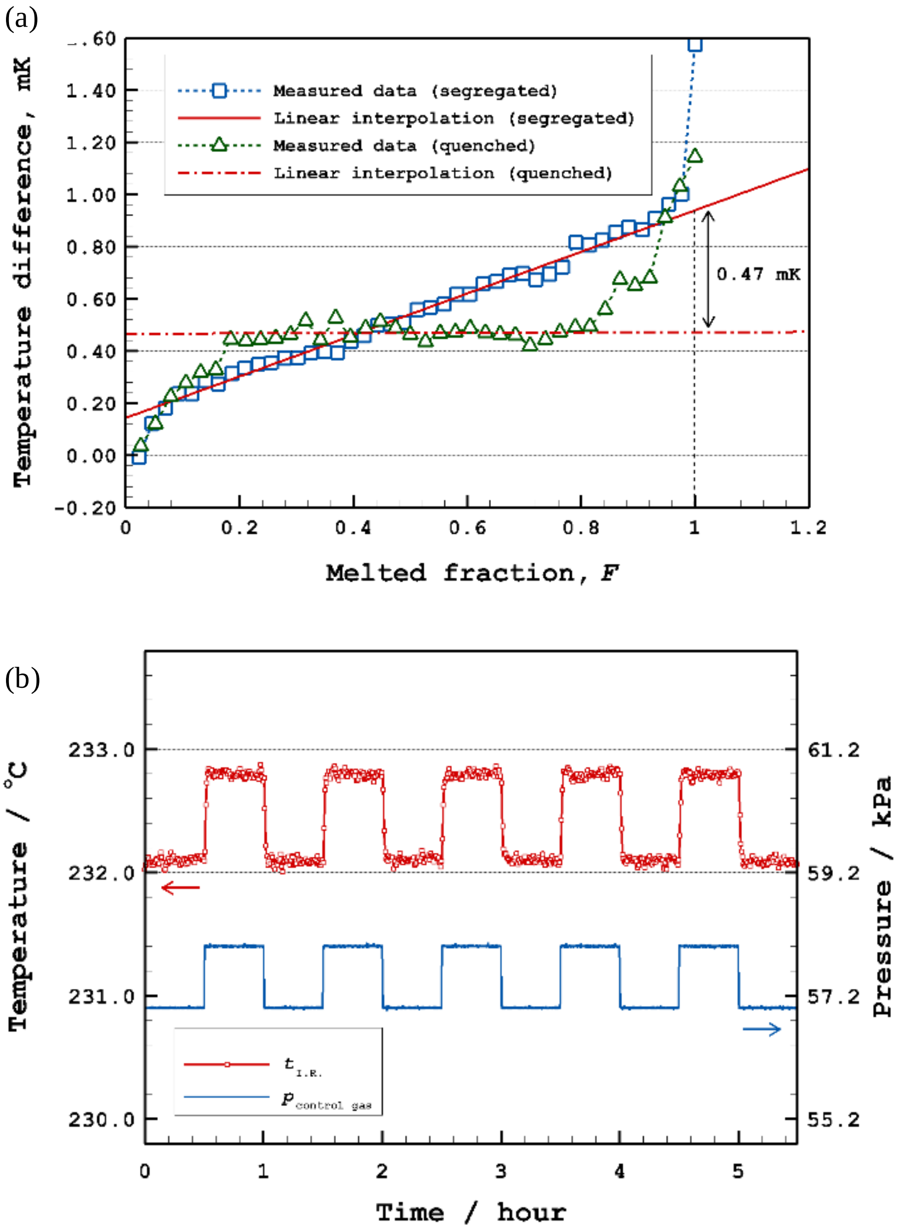

Figure 19. (a) Generated steps of the isothermal region temperature to the square wave-type pressure changes of the control gas; (b) Melting temperature variations of the segregated and quenched samples in terms of the temperature difference from the outside-nucleated freezing temperature of tin [49].

### 4.8 National Metrology Institute of Japan (NMIJ).

A comparison measurement system was developed in the late 1990s at the NMIJ, to investigate the non-uniqueness of the International Temperature Scale of 1990 (ITS-90) in the range from 65 °C to 157 °C. A special design was made for this experiment, involving a water filled GCHP, equipped with an internal copper comparison block, with seven thermometer wells, acting as an isothermal chamber [53]. The length of the block is 200 mm and the diameter of each well is 9 mm. The centre thermometer well No. 7 is placed vertical, while the other wells are slightly leaning and converging towards the bottom. Ceramic fibre and aluminium disks are inserted above the copper block to reduce the vertical heat flow. In the present study, the comparison measurements were performed on six SPRTs inserted into wells No.1 to No.6. A temperature uniformity within

0.05 mK is obtained over a zone of 10 cm from the bottom. This result suggests that a high temperature uniformity is obtained in the comparison block in the vertical direction and that vertical heat loss is negligibly small. The uncertainty due to the thermal stability was evaluated to be within 0.034 mK. Preliminary measurements of the non-uniqueness were performed using six standard platinum resistance thermometers of the same model. The non-uniqueness was evaluated with uncertainty of 0.08 mK, in terms of sensitivity of the overall system to detected minimum differences among the tested thermometers.

Having achieved valuable results involving water as working fluid, staff at NMIJ developed a further system, scoring the record of the biggest GCHP ever used in metrology: the length being 1100 mm and the outer diameter 48 mm [54]. The furnace was newly developed using a stainless steel heat pipe, equipped with two thermometer wells and used for the evaluation of inhomogeneity of thermocouples. One well was used to host the thermocouple under test and the second one for an industrial platinum resistance thermometer (IPRT), used to monitor the temperature of the heat pipe or to measure the temperature profile along the well. The performance of this system was more than satisfactory for the purpose: 0.5 mK of temperature stability and ±1 mK of uniformity over a vertical zone of 600 mm.

The same group at NMIJ also developed and used a sodium GCHP as a high precision furnace for primary thermometry. [55]

## 5 Present work and future perspectives.

Together with the new definition of the kelvin adopted in November 2018 with the resolution 1 of the 2018 by the *Conférence générale des poids et measures* (CGPM - General Conference on Weights and Measures), the ITS-90 is now the subject of improvement and studies. It will be open to broader and more flexible ways for the "*mise en pratique*" of the unit, i.e. the realisation of the temperature scale which approximates the unit, and to new definitions in specific fields of interest, including direct measurements of thermodynamic temperature and relationships, as adopted by the *Consultative Committee for Thermometry* since 2011.

At present, since no temperature points are defined between the fixed points of aluminium and silver, corresponding to the wide temperature interval between 660 °C and 962 °C, studies are being carried out for the proposal of a new standard in this temperature range.

In February 2004, a Euromet project was discussed and proposed in agreement between nine European National Metrology Institutes [56]. The project started an EU cooperative work and raised widespread interest on the use of GCHPs and the temperature amplifier as a new temperature standard in platinum resistance thermometry. Since then, several systems and devices have been studied, developed, manufactured, and assembled at INRiM. New GCHPs have been manufactured building on the experience with these devices acquired in the past years. An innovative pressure controller has been studied and built for the purpose; the relative electronic components have all been developed and made by INRiM. Dedicated furnaces have been designed and assembled, together with all the proper cooling lines. Control and acquisition software has been developed at INRiM too. Some of those components have been tested and characterized both at the Italian and French Institutes of Metrology LNE-INM/CNAM. In 2011, on the basis of this cooperation, a proposal was submitted under the "SI-Broader Scope" call of the European Metrology Research Project of Euramet, the association of European National Institutes of Metrology. The proposal was successfully evaluated and became a work package of the SIB10 NOTED Joint Research Project on novel techniques for traceable temperature dissemination. The work started in June 2012 with two main deliverables: a new extended study of the wideness of the non-uniqueness of HT-SPRTs and thermocouples and a proposal for a possible practical reference function for the calibration of contact thermometers between the Al and Ag fixed points and a device for temperatures between the Al and Ag fixed points. Results of an extended study on ITS-90 non-uniqueness have been published by Coppa and Merlone in 2016 [20], where a possible function linking lower temperature of Hg phase transition to higher temperature of Na is calculated and was based on the findings on the Hg-Na relationship extended to lower and upper pressures.

Based on the results of the NOTED project [57], a further proposal was prepared and presented as a task of the EMPIR joint research project proposal “Real-K”, started in September 2019. The proposed work will improve our current understanding of Type 3 non-uniqueness [20, 58], establishing upper limits for this kind of uncertainty reduced by at least 30 % with respect to those available in the literature), from a set of high-quality comparison measurements performed in different laboratories The analysis of the measurements will be coordinated by NPL, which has recently proposed a new method for processing the data, that permits isolation of the Type 3 non-uniqueness signal, nearly completely "clean" of other effects. Taking advantage of the high temperature stability that can be obtained using GCHPs, INRiM will perform the investigation on Type 3 non-uniqueness by comparing a batch of different HTSPRTs in the temperature range 660 °C to 960 °C, thus improving the results published by Coppa and Merlone in 2016 [20].

The “Temperature Amplifier” here described could, in principle, be used to realize a possible future temperature scale. Any accurate reproducible temperature range may be used to realize another temperature range, since the relationship between the two ranges is thermodynamically defined by the two pure substances used for the vapour liquid transitions. By realizing the temperature range between aluminium and silver freezing points with a Temperature Amplifier, HTSPRTs could be calibrated very accurately over the whole temperature range. The main advantages of choosing such an approach are the elimination of non-uniqueness due to HTSPRTs and the limited reproducibility of the temperature calibration on the maximum required operating range with more calibration points available in between the ITS Al and Ag fixed points.

The experiments on temperature amplification and the connected thermodynamic relations have been carried out with very high accuracy. For example, starting with an accuracy better than about 2 mK in the mercury phase transition, the amplification system can yield an accuracy of about 4 mK in the sodium HP temperature range. This last value is 2 to 3 times better than the corresponding values obtained by interpolation of the HTSPRT calibration between the aluminium and the silver freezing points (HTSPRTs are affected at high temperature by large calibration drifts, instability and problems related to the non-uniqueness of the temperature scale). Other HPs can be connected to the pressure controlled line, in order to investigate other relations in different temperature and pressure regimes. The temperature amplification technique represents significant innovation in temperature metrology. Techniques to disseminate thermodynamic realisation of the temperature scale will be subject of growing interest also in line with the newly adopted definition of the kelvin.

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