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An adiabatic calorimeter for calibrating capsule type thermometers (CSPRTs) in the range 54 K to 273 K

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Abstract. A new comparison system has been constructed for the calibration of capsule-type standard platinum resistance thermometers (CSPRTs) below 273.16 K at the National Institute of Standards (NIS-Egypt). The system can compare two CSPRTs at once. A gold plated comparison block, in which CSPRTs are mounted for calibration, is made from oxygen-free high-conductivity copper. A description of the system and the comparison block along with the comparison method is given. The standard uncertainty related to the temperature control of the system is estimated to be 0.04 mK. The calibrated values for CSPRTs and obtained using the comparison system are in good agreement with those obtained by the direct realization of the low temperature fixed points of the ITS-90 within the combined standard uncertainty.

Keywords: CSPRTs; ITS-90; adiabatic calorimeter; fixed points; uncertainty

1 Introduction

The National Institute for Standards (NIS)-Egypt, in cooperation with the French National Metrology Institute (LNE-CNAM)-France, has developed an adiabatic calorimeter for the realization of the International Temperature Scale of 1990 (ITS-90) [1] at the highest level of accuracy in the range from 54 K to 273 K.

NIS has made use of the experience of the LNE-CNAM [2–5] to build the calorimeter that would work with the existed facilities.

In this cooperation project, the device was intended to realize the triple points of oxygen (54.3584 K) and argon (83.8058 K), used in the definition of the ITS-90 below 0 °C. Realization of these fixed points is performed by Capsule Standard Platinum Resistance Thermometer (CSPRT). The methods of realizing these temperature fixed-points involve complex experimental techniques and procedures, besides it consume significant time.

So as a first step, the present work presents a comparison system using the adiabatic calorimeter. It comprises a comparison block, made from oxygen-free high-conductivity copper, in which CSPRTs are mounted for calibration. The adiabatic calorimeter accommodates the block instead of the fixed-point cells. The techniques and methods for attaining stable temperatures with the lowest possible drift are simple as will be explained in the following sections. Thus, the time and cost of calibrating CSPRTs can be reduced using this comparison system.

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Performance of the system will be checked also by investigating the differences between calibration values obtained using the comparison system and those obtained by realizing the low-temperature fixed points directly.

2 Experimental arrangements

Figure 1 shows a schematic side view of our new comparison cryostat for the calibration of CSPRTs connected to the controlling and measuring equipment.

The copper block is cylindrically symmetric and has four wells for CSPRTs in the annular region. The CSPRTs are liberally coated with vacuum grease during insertion into the wells to enhance their thermal contact with the block. Electrical leads are thermally anchored at top flange of the inner copper-can of the cryostat. The comparison block is enclosed in an isothermal copper-can to maintain a uniform thermal condition and to avoid the effect of radiation from the outside. The top-flange of this copper-can is fixed to an outer vacuum-can through three stainless steel rods and four brass rods. A Pt-100 sensor is fixed to this flange to monitor and control the temperature of the thermal shield through a PID regulation.

The calorimeter is connected to two vacuum systems; the first system is connected to the main central tube which leads to the experimental space that containing the copper block. The system consists of a rotary pump associated with a diffusion pump. With this pumping of better than 5×10^{-4} mbar, a good thermal isolation could be maintained.

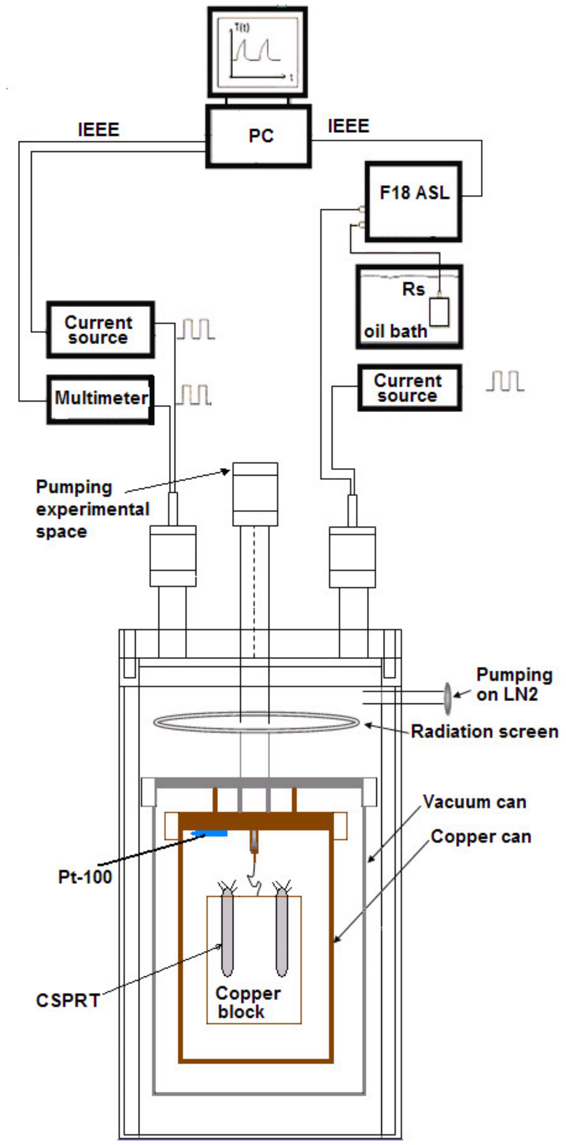

Fig. 1. Schematic side view of the comparison cryostat.

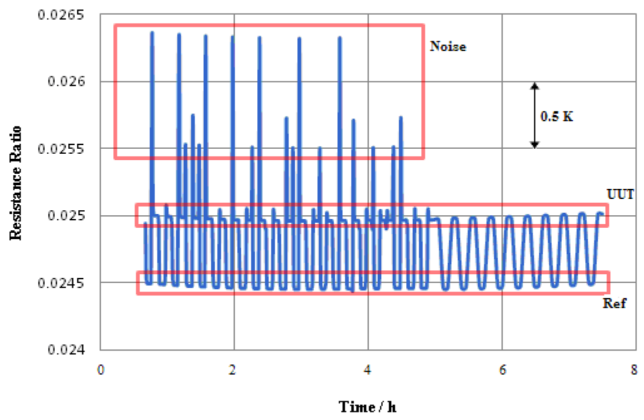

Fig. 2. Controlled temperature near the triple point of O_2.

The second vacuum system is a highly efficient rotary pump BUSCH model "vane vacuum 0100". The pump is connected to the space above the liquid nitrogen. This pump was chosen such that it has the capability to pump the continuous flowing of cold vapor-nitrogen to the extent that having very low temperatures of solid nitrogen lower than 54 K.

A helium gas was used when there was a need to have an exchange gas in the experimental space that helps in adjusting and stabilizing the temperature of the copper block to certain values.

The PID controller controls an electric heater fixed on the top flange of copper-can. A Keithley-224 current source was used to pass the electric current to this heater. The controller measures the temperature by using A Keithley-2000 multimeter used to measure the resistance of the Pt-100. The current source was adjusted to work within the range from 0 to 70 mA. An increase of the resistance of Pt-100 means an increase of the thermal-shield temperature. The current source wtextas controlled such that it did not exceed 70 mA to avoid burning of manganin heater. The PID controller was derived through a software working under LabView environment. The temperature of the isothermal copper-can is controlled just below the comparison-block temperature using a temperature controller through the Pt-100. The temperature difference between the comparison-block and the isothermal-shield is about 0.25 K.

Measurements of CSPRTs (reference thermometer "Tinsley type 5187L SN. B300" and an under test thermometer "Tinsley code UUT") were obtained using an F18 ASL resistance bridge in conjunction with a 100 Ω Tinsley standard resistor maintained in a thermostated oil bath at 20 °C. The reference thermometer is calibrated according to the ITS-90 definition. This thermometer was chosen after showing a good stability of less than 0.1 mK over several years at the triple point of water.

3 Results and discussion

Regulation operation was started to control the temperature of the copper can at temperatures close to the triple points of oxygen, argon, and finally mercury. This process was working automatically under the control of the PID.

Measurements of the resistances of CSPRTs (reference and UUT) were measured using the resistance bridge through data acquisition software that manage measurement of the reference CSPRT and then switches to the UUT CSPRT. The program allows monitoring and collection of data on a datasheet file for further analysis.

Figures 2 and 3 show typical results of the measured resistance ratios of both of reference and UUT CSPRTs, at temperatures near the triple points of O_2 and Ar. The variation in temperature between holes of the block are determined by switching the positions of two thermometers at double runs and measure the difference in temperature

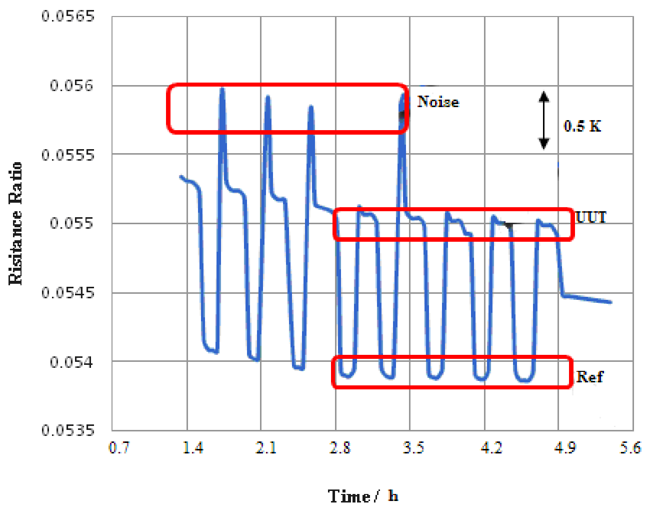

Fig. 3. Controlled temperature near the triple point of Ar.

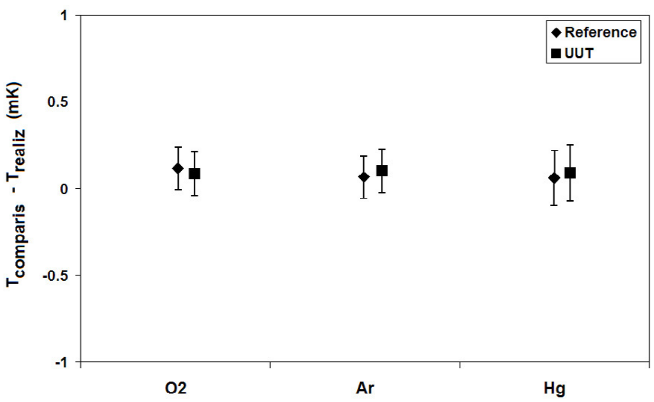

Fig. 4. Temperature difference between calibrate values by comparision and those calibrated by realization of O_2TP, ArTP and HgTP.

to finally calculate temperature gradient. It was found to be 0.04 mK.

To check the performance of the comparison technique, the difference between calibration values of CSPRTs obtained with the comparison system and those obtained by realizing the triple points of O_2, Ar, and Hg have been investigated with the similar way as given by [6]. Figure 4 shows the difference between the calibration values obtained with the comparison system and those obtained by the direct realization of the low-temperature fixed points.

The error bars shown in the figure represent the combined standard uncertainty for the calibration using the comparison system as shown in Table 1.

The calibrated values of CSPRTs obtained with the comparison system are in good agreement with those obtained by the direct realization of the temperature fixed points of the ITS-90 within the combined standard uncertainty.

Table 1. Uncertainty budgets for the calibration of CSPRTs at temperatures close to fixed points using the comparison system.

Uncertainty component (°C)	Value (mK)		
	O_2	Ar	Hg
CSPRT short term stability	0.05	0.05	0.05
Bridge measurement	0.01	0.01	0.01
Temperature control	0.04	0.04	0.04
Self-heating error	0.01	0.01	0.01
Spurious heat flux	0.04	0.04	0.04
Temperature instability	0.03	0.03	0.03
Temperature uniformity	0.04	0.04	0.04
Fixed point realization	0.04	0.06	0.15
Standard combined uncertainty	0.10	0.11	0.18

4 Conclusion

A new comparison system has been constructed and developed for the calibration of CSPRTs at NIS-Egypt using adiabatic calorimeter with improved temperature stability of the copper comparison block. It has been confirmed that the temperature of the comparison block is stable within 0.06 mK at temperatures near the temperature fixed-points especially at triple point of oxygen, argon and mercury. Furthermore, the time and cost of calibrating CSPRTs can be reduced using a comparison system that employs a pumping technique over liquid nitrogen in comparison to the time and cost for the direct realization of the temperature fixed-points of the ITS-90.

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