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New temperature reference cells for calibrating CSPRTs under adiabatic conditions

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Abstract. Two new miniature metallic sealed-cells containing the triple point of water, WTP (273.16 K) and the triple point of mercury, HgTP (234.3156 K) have been constructed for the realization of the International Temperature Scale of 1990 (ITS-90) at the National Institute of Standards (NIS-Egypt). The new cells will provide facilities for the calibration of Capsule-type Standard Platinum Resistance Thermometers (CSPRTs). The two cells will complete a multi-cells compartment in order to calibrate CSPRTs in one single experiment. The multi-cells compartment contains two other cells; the triple point of argon (83.8058 K) and the triple point of oxygen (54.3584 K) that has been already developed and characterized before [M.G. Ahmed, Y. Hermier, Development of an adiabatic calorimeter in the range 54 K–273 K in frame of a scientific collaboration LNE-NIS, AIP Conf. Proc. **1552**, 153 (2013)]. The system can calibrate two CSPRTs at once. The compartment is accommodated in an adiabatic calorimeter. With a special technique, the calorimeter could go down to a temperature of 52 K using liquid nitrogen and special pumping system.

Keywords: CSPRTs, ITS-90, multi-cells, fixed points, uncertainty

1 Introduction

The National Institute for Standards (NIS)-Egypt, in cooperation with the French National Metrology Institute (LNE-Cnam)-France, had developed multi-cells compartment [1, 2] containing two cells; the triple point of argon "ArTP" (83.8058 K) and the triple point of oxygen "O_2TP" (54.3584 K), for the realization of the International Temperature Scale of 1990 (ITS-90) [3].

NIS decided to complete the multi-cells compartment with another two new miniature cells, of triple point of water "WTP" (273.16 K) and triple point of mercury "HgTP" (234.3156 K), in order to have the temperature range from O_2TP up to WTP in on single experiment. This in turn will improve the calibration uncertainty over the concerned whole temperature range in only one system (calorimeter with multi-cells) instead of using different systems (calorimeter and other realizing systems for WTP and HgTP).

In addition, the used methods of realizing these temperature fixed-points involve complex experimental techniques beside it consume significant time.

2 Miniature cells

2.1 Choice of material

For the WTP cell, taking the advantage that copper has a high thermal conductivity and with copper oxide-layer formed due to interaction with water, the layer will prevent water contamination from pure copper [4]. Hence, a water cell with a copper wall that is oxidized before performing the final filling and sealing can remain stable with time.

For the HgTP cell, stainless steel was selected because it has no chemical interaction with mercury [5].

2.2 Design of the cells

The geometry of WTP cell, as shown in Figure 1, was designed to be integrated with the multi-cells compartment, thus to have a shape similar to those of O_2TP and ArTP cells. This enables studying the thermal resistance between the sensing element of the CSPRTs and solid-liquid interfaces temperatures of the substances within the cells [6].

The HgTP cell was designed, as shown in Figure 2, to be very close to the CSPRT shape [5,6]. The two new cells assembled with ArTP and O_2TP to form a final multi-cells compartment as shown in Figure 3.

2.2.1 Water triple point cell

The main criterion for the design of the cell is the ease of cleaning and filling. The cell has an outer diameter of 55 mm and height of 14.5 mm. The cell is equipped with two filling tubes of 25 mm length; this allows cleaning of the cell with a continuous water flushing flow to

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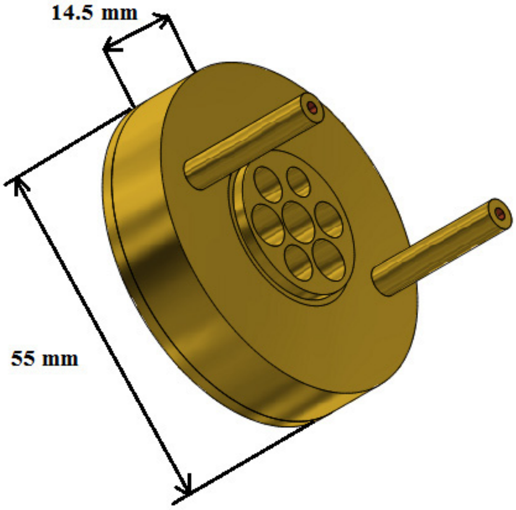

Fig. 1. WTP cell design.

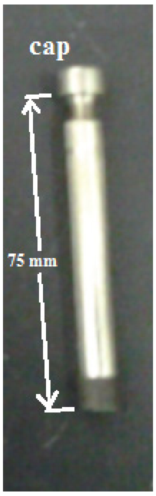

Fig. 2. HgTP cell design.

remove the impurities and develops the oxide layer inside the cell. The centre of the cell contains several holes with various diameters to accommodate two CSPRTs, HgTP cell and central hanging rod.

2.2.2 Mercury triple point cell

The cell was made of stainless steel tube with an outer diameter of 6 mm and a height of 75 mm. The cell has a cap which is a threaded screw fixed at the top edge with a leak of 0.2 mm when closed to achieve a triple point state during pumping the space around it. The bottom of the tube was epoxy sealed.

2.3 Cleaning of the cells

2.3.1 WTP cell cleaning

The WTP cell was cleaned using the same technique that have been used by [4] by first using a continuous flushing

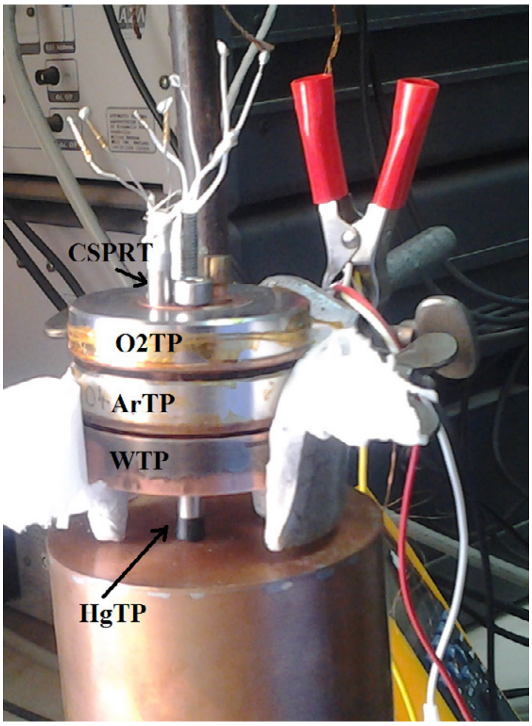

Fig. 3. Multi-cells compartment.

with double-distilled water flow, then filling with water for one day, and then backed in drying oven for another day.

The cell was washed with alcohol, formaldehyde and distilled water, then backed in a dryer for one day.

The cell was flushed with water steam system for two weeks.

2.3.2 HgTP cell cleaning

All parts of the HgTP cell were fully immersed inside formaldehyde for two days then dried for one day. They were then immersed inside acetone for one day, the cell parts are baked inside a drying oven for one day. Afterwards, the cell parts were cleaned inside an ultrasonic bath for 30 min.

Finally the cell parts were dried inside the drying oven for one more day.

2.4 Cell filling

2.4.1 WTP cell filling

The flushing process of double-distilled water lasted up to two weeks should be enough to develop a suitable oxide layer on the inner wall of the cell. Sealing was performed at one filling tube so that only liquid water and water vapour are contained inside the cell. The cell was completely filled with water, and then it was heated slightly below the water boiling point to increase the vapour pressure of the water. Vapours inside the cell are evacuated via the filling tube; this avoids external air entering the cell. With this technique, by allowing a slow flow of water vapour and weighing the cell in the same time a sealing of the filling tube by pinching was performed with the required mass of water sample inside.

The amount of water inside the cell was 11.7 g which is enough to obtain a sufficient plateau according to the amount of heat needed and latent heat of fusion of water 334 J g^{-1} for the phase transition [6,7].

2.4.2 HgTP cell filling

Pure sample of mercury of 99.99999% was used with an amount of 8.6 g.

Filling was performed by pouring the sample directly into the cell in small drops with a little agitation of the cell body.

3 Experimental arrangements

At the beginning, the CSPRTs that were intended to be used in characterizing the new cells, were inserted in the laboratory reference cells. These cells are large cells used to maintain the ITS-90 [8] and for calibration of long-stem SPRTs. An alcohol bath "Fluke Model 7381" with a stability of 0.006 °C was used for the realization of HgTP. Another alcohol bath "Hart Scientific Model 7012" with a stability of 0.0008 °C was used for the realization of WTP.

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 23 °C. The reference thermometer is calibrated according to the ITS-90. This thermometer was chosen after showing a good stability of less than 0.1 mK over several years at the triple point of water and mercury.

The next step is to mount the new cells with the multi-cells compartment and insert it into the adiabatic calorimeter. The adiabatic calorimeter is described in details in references [1,2].

Figure 4 shows schematically the experimental setup of measurements using the adiabatic calorimeter.

It contains the calorimeter that enclosing the multi-cells compartment. Connections of the controlling and measuring equipment are shown.

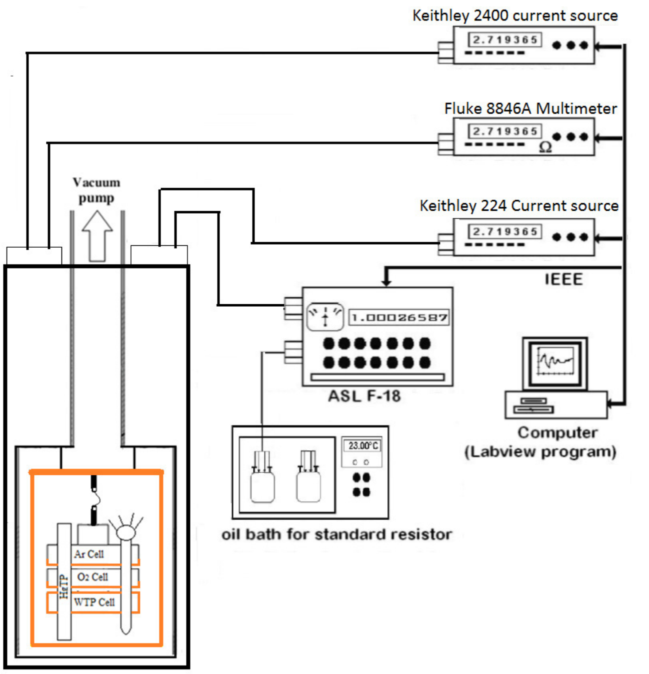

Fig. 4. Schematic view of the experimental setup using the adiabatic calorimeter.

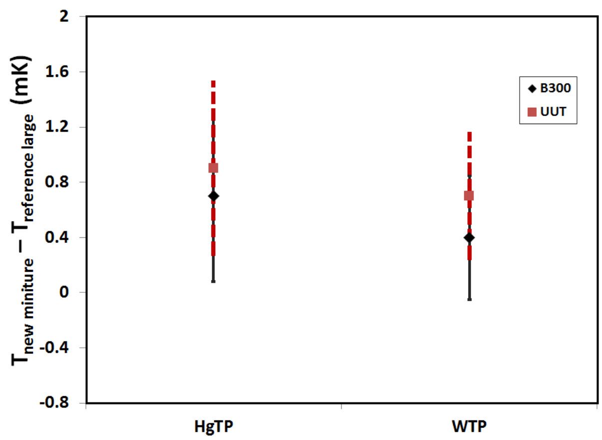

Fig. 5. Temperature differences between new miniature cells and laboratory reference large cells.

4 Results and discussion

Actually, 7 and 21 plateaus were realized for both of WTP and HgTP respectively in large cells in order to obtain reliable calibrations for the two CSPRTs. The fixed-point temperature of the thermometers showed good stability within 1 mK over all runs.

For the new miniature cells, only 2 runs were performed in the adiabatic calorimeter. Figure 5 shows the temperature differences for both of the two new miniature cells with those of the laboratory reference cells. The differences were calculated using the average values obtained for each.

5 Conclusion

New WTP and HgTP cells have been constructed and developed for the calibration of CSPRTs at NIS-Egypt using adiabatic calorimeter with improved temperature stability.

The characteristics of new metallic miniature cells for the water and mercury triple points have been discussed and preliminary results have been presented. The realizations of fixed point in miniature cells show temperature differences for HgTP of 0.7 and 0.9 mK using CSPRTs B300 and UUT, respectively. It shows differences of 0.4 and 0.7 mK for WTP. The results are very encouraging, but more measurements are needed using the adiabatic calorimeter to test the reproducibility and the stability of the phase transitions.

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