BIPM Capacity Building & Knowledge Transfer Programme

2021 BIPM - TÜBİTAK UME Project Placement

REPORT

Project Name	Study of stability of HTSPRT at Silver (Ag) and Aluminum (Al) fixed points
Description	Calibration of HTSPRT in Silver (Ag) and Aluminum (Al) Fixed Point cells, Stabilization of HTSPRT in WTP cell and calculation of the measurement uncertainty.
Author, NMI	Iska Kolaveri , DPM, Albania
Mentor at TÜBİTAK UME	Narcisa ARIFOVIÇ , Temperature Laboratory, TÜBİTAK UME, Turkey
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Motivation & Introduction

The BIPM-TUBITAK UME project placement 2021 in thermometry field aimed to realize the calibration process of SPRT according to ITS-90 at high temperatures, at two fixed points, Silver Ag FP (961.78 °C) and Aluminium Al FP (660.323 °C), the progress of the HTSPRT during the calibration, and also the control in WTP is always necessary to see the stability of HTSPRT. Usually the order of development of the process is the realization of Water Triple Point before and after the realization of the Fixed Point cell.

For the HTSPRTs that are calibrated at the triple point of water (0.01°C) and at the freezing points of Aluminium (660.323 °C) and Silver (961.78 °C), the deviation function is defined by ITS-90 as:

$$W(T_{90})-W_r(T_{90})=a[W(T_{90})-1]+b[W(T_{90})-1]^2 + c[W(T_{90})-1]^3 +d[W(T_{90})-W\ (660.323\ °C)]^2$$

With the values a, b, c and d being obtained from measurements.

During my stay at UME, I learned in general about the realization of all fixed points, and specifically the 2 fixed points Ag FP and Al FP as it was proposed at the beginning of this project.

The main objectives of this project are:

- The realization of the ITS-90 fixed points (Ag and Al fixed points).
- Improvement of measurements for studying the stability in higher temperature of SPRTs at Ag and Al Fixed points.
- Check and control in WTP.
- The determination and calculation of the components for uncertainty budget.

Research

In this project are taken as a case study two different types of SPRT: SPRT 0.25 Ohm at 10 mA and HTSPRT 2.5 Ohm at 5 mA. They are taken in order to study their stability behavior for calibration at high temperatures.

Initially, the control measurements were performed at the water triple point cell to see if the HTSPRTs meet the stability criteria according to ITS-90. Measurements were taken via Resistance Bridge for each of the HTPRTs in two different currents for a period of 30 minutes without disconnection to check the thermometers self-heating. One HTSPRT resulted to be stable after the check, the other one didn't meet the criteria according to ITS-90, and it was necessary to carry out another process called "annealing".

Annealing is a heat treatment process which alters the microstructure of a material to change its mechanical or electrical properties. Typically, annealing is used to reduce hardness, increase ductility and help eliminate internal stresses to make it more workable.

This process was repeated 3 times, until the SPRT reached the allowed correction of 3mK.

- The following is the process of characterizing fixed points:

For realizing the *fixed point of Silver*, it should be melted in the furnace overnight at about 5°C above the freezing point temperature. One control HTSPRT must be inside the cell to measure and control its temperature overnight. The next day, after we have set the furnace temperature to cool down in order to initiate freezing of the fixed point metal and continue monitoring of the cooling of the metal via control thermometer. After that we have to perform immediate cooling by inserting the cooling rod into the fixed point cell for 1 minute, then to wait for 1 hour to perform the measurements.

The calibrated HTSPRT is placed in another furnace for preheating at 550°, after 30 minutes we place it inside the fixed point and wait until it stabilizes. The HTSPRT is connected to a resistance bridge, which is connected to a computer with the relevant software. Measurements are performed in different currents to see self-heating of the HTSPRT. After the completion of the measurements at this fixed point, next day the measurements are performed again at the triple point of water, for the non-repeatability and the stability of HTSPRT.

Figure 1. Preheating of HTSPRT

Realization of the *fixed point of Aluminum*.

Process almost the same as the fixed point of Silver cell.

The furnace temperature is set at 680 °C, we wait for its stabilization for 45 minutes, and for realizing the freezing of the fixed point cell we placed inside that cell a rod from room temperature for 1 minute. Meanwhile, we placed the HTSPRT for 30 minutes in another furnace at a temperature of 550 °C to realize preheating. Then we place the HTSPRT inside the fixed point and wait for stabilization for 45 minutes (to reach freezing plateau), then we take automatically the measurements by the relevant software in different currents.

The whole process of characterization of fixed point cells, as well as the calibration process is performed under the guidance of the mentor of the Temperature Laboratory of TÜBİTAK UME.

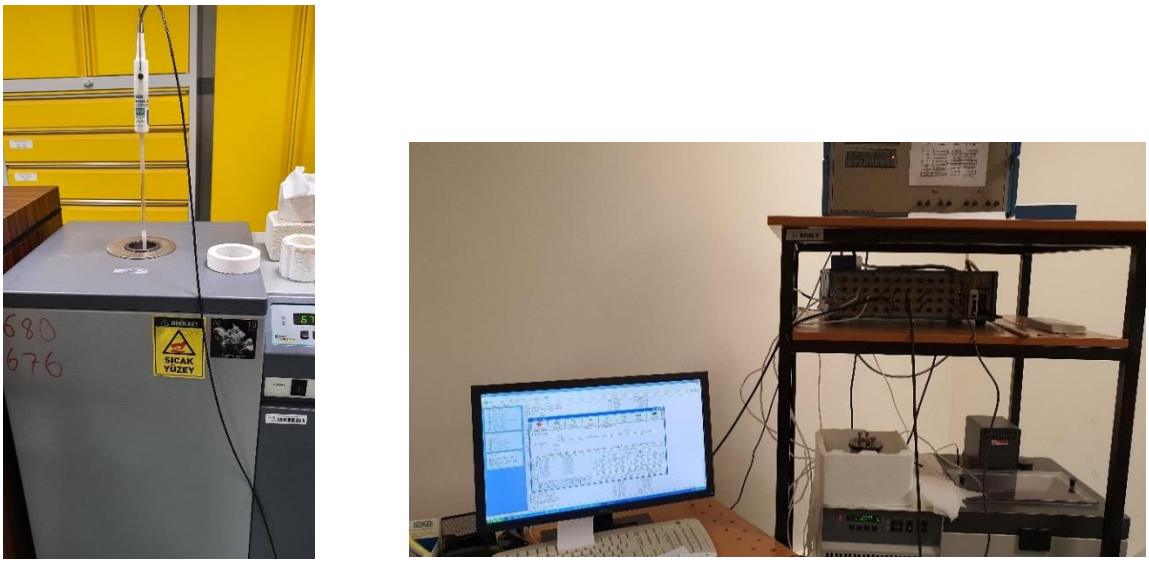

Figure 2. a) Calibration of the HTSPRT in Al Fixed point. b) Connection with thermometric bridge and measurements taken from software.

After completing the measurements, a series of accurate calculations are performed, of the measured resistances, as well as using the functions of the respective equations for calculating the temperature. Also taking into account all the components for calculating the uncertainty budget, for calibration at Water Triple point and in fixed point cells. As an example the components of the uncertainty budget for calibration in Water Triple point are described below:

1- Type A method
2- Type B method:
   - Electrical measurements
   - Self-heating
   - Spurious effects on the SPRT
   - Hydrostatic effects
   - Heat losses
   - Chemical impurities
3- Combined uncertainty (at 1 standard deviation level)

This uncertainty at the triple point of water is also taken into account in calculation of the uncertainty for calibration of SPRTs and HTSPRTs at fixed point cells.

Conclusions and Future Work

Participation in "BIPM - TÜBİTAK UME project placements" allowed me to improve my technical skills and build knowledge towards the thermometry field.

This project was helpful for me and it will be a great contribution to my institution, to put into operation the primary laboratory, to use the fixed points that we possess.

These knowledge and skills will assist me to update our technical procedures and calculation of uncertainty for HTSPRT calibration in fixed point cells, in contact thermometry. From the experience gained through my studies, I will produce a project plan to participate in an interlaboratory comparison and to submit CMCs in the future.

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