TEMPERATURE NATIONAL STANDARD MODERNIZATION THROUGH SOFTWARE MONITORIZATION OF THE INDIUM FREEZING PLATEAU REALIZATION PROCESS

Cosmin DINU[1], Dumitru Marius NEAGU[2]

This paper presents the results obtained during SPRT (Standard Platinum Resistance Thermometer) fixed point calibration at the indium freezing point (156,5985 °C) using national standard equipments from the INM (Institutul National de Metrologie) including a newly developed Test Point software used for the indium freezing plateau realization process monitoring. It also highlights the advantages of using a monitoring instrument for the realization process of SIT 90 temperature fixed points for the present and future studies. These studies will lead to a better SPRT (Standard Platinum Resistance Thermometer) fixed point calibration uncertainty.

Keywords: temperature, fixed points, calibration, software

1. Introduction

The International Temperature Scale of 1990 (ITS-90) is based on a series of fixed points. At temperatures above 273.16 K, the freezing points of indium, tin, zinc, aluminum, silver, gold, and copper are the defining fixed points of the ITS-90. The temperature value of the indium freezing point is 156,5985 °C. Most of the fixed points are the freezing points of specified pure metals. Pure metals melt and freeze at a unique temperature through a process involving the absorption or liberation of the latent heat of fusion.

Primary fixed points are defined in the International Temperature Scale of 1990 (ITS-90) [1]. In the field of temperature standards, fixed points play an important role, from the definition of the temperature scale to its dissemination.

Under controlled conditions these freezing points are highly reproducible. A metal freezing point is the phase equilibrium between the liquid phase and solid phase of a pure metal at a pressure of one standard atmospheric pressure (101,325 Pa).

[1] Eng. Temperature Group, National Metrology Institute Bucharest, Romania
[2] PHD Eng., Temperature Group, National Metrology Institute Bucharest, Romania

2. Equipments used for the Indium Freezing Plateau Realization Process

In order to obtain the indium freezing plateau the following temperature national standard components are used. One of these is a fixed point sealed cell that contains pure In. This cell is placed in an Isotech Ayries fluidized calibration furnace, that has a Temperature Range from 50°C to 700°C. The calibration furnace is supplied with RS232 serial interface.

Another component is the F18 primary standard thermometry bridge. The bridge is also supplied with RS232 serial interface. The F18 uses a reference resistor introduced in a water bath that is maintained at a constant 20 °C temperature during the calibration process. The influences of the reference resistor are not included in the present paper.

A four wire 5699 High Temperature Metal Sheath SPRT is connected to the bridge. The SPRT measures the temperature inside the indium cell during the whole calibration process. It has a nominal resistance value for the triple point of water of 25.5 Ω (±0.5Ω), and a temperature range from -200 °C to 670 °C. According to SIT 90, this SPRT can be calibrated at 8 fixed points: from Ar (triple point: -189,3442 °C) to Al (freezing point: 660,323 °C).

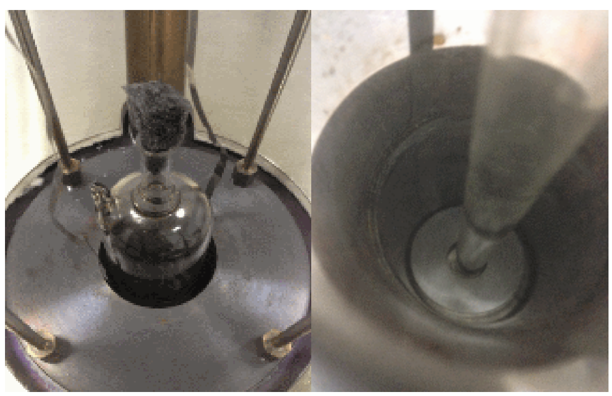

Fig. 1. Fixed point sealed cell that contains pure In

The F18 primary standard thermometry bridge and the calibration furnace are connected to a PC by the RS232 interface.

(1) F18 primary standard thermometry bridge

(2) The calibration furnace is supplied with RS232 serial interface; contains the indium cell

(3) The four wire 5699 High Temperature Metal Sheath SPRT

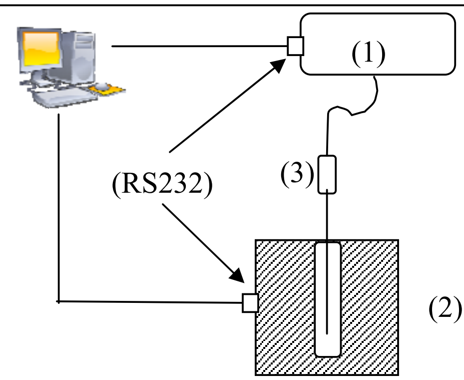

Fig. 2. Equipments used for the Indium Freezing Plateau Realisation Process

3. Software Configuration

As mentioned earlier the PC is connected to the F18 primary standard thermometry bridge and the calibration furnace. It has installed a Test Point software used for the indium freezing plateau realization process monitorization. The software provides a window that contains configuration fields that are specific for different calibration components and different fixed points.

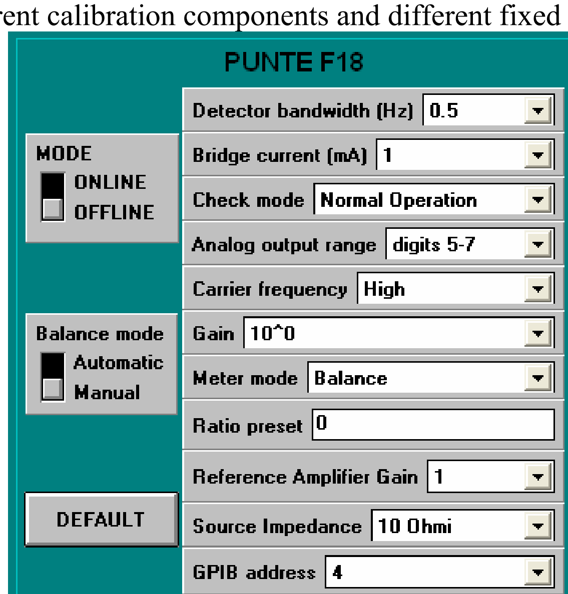

Fig. 3. Bridge specific parameters for the indium freezing plateau realisation process

In the configuration window the user can choose the type of primary standard thermometry bridge and the calibration furnace. Here the user can also set the bridge specific parameters for the indium freezing plateau realization process.

In this step, the software also allows the user to (remotely) set the furnace temperature before the process actually starts, as well as the data acquisition rate. With the program the user sets the measurement configuration then launches the freezing plateau realization process. The program monitors the temperature inside the furnace, as well as the resistance values of the SPRT and sets the furnace temperature according to these values.

4. Indium Freezing Plateau Realisation Process Necessary Steps

In the next section are described the necessary steps in order to achieve the In freezing point. First, the furnace temperature is set to 165 °C (above the indium freezing point). The fixed point cell temperature increases so the SPRT resistance values will increase as well. When the SPRT resistance values begin to stabilize (around 41,043 **Ω**) the indium is starting to melt. Than the furnace temperature is set to 160 °C. When the indium ends its melting plateau the SPRT resistance values begin to increase [3].

At this moment the furnace temperature is set again to 165 °C in order to completely melt the indium inside the fixed point cell. The furnace can be let overnight with this value. Than, after the indium inside the fixed point cell is completely melted, the furnace temperature is set to 155 °C (below the indium freezing point - 156,5985 °C).

Finally, after a decalescence the freezing point is obtained.

5. Indium Melting Plateau Realization Process Measurement Results

In the next section the above procedure implementation measurement results are described.

The following data is obtained during the process for an acquisition rate of 15 seconds. The data is saved for further analysis and improvement. The most relevant fields for the present study are a. time, b. SPRT resistance values and c. furnace temperature.

First the melting process is analyzed. The following graphic illustrates data obtained for 11 hours. At the beginning of the process the furnace temperature is 165 °C (the pink chart – axis on the right). From this point the fixed point cell is heating, so the SPRT (that is placed inside the cell) resistance values increase (the blue chart – axis on the left). This trend stops when the SPRT resistance value is about 40.04 Ω.

When this happens, the furnace is set to 160 °C. Next, the indium inside the fixed point cell forms a melting plateau for about 4 hours. This means that SPRT resistance value stabilises at 40.04 Ω (figure 5 shows these values in detail). The furnace temperature is kept constant until the end of data acquisition. Observing the chart in figure 5 the indium inside the fixed point cell formed a melting plateau between 40.043 Ω and 40.044 Ω. Figure 5 contains data from figure 4 (SPRT resistance value chart) showing only the melting plateau.

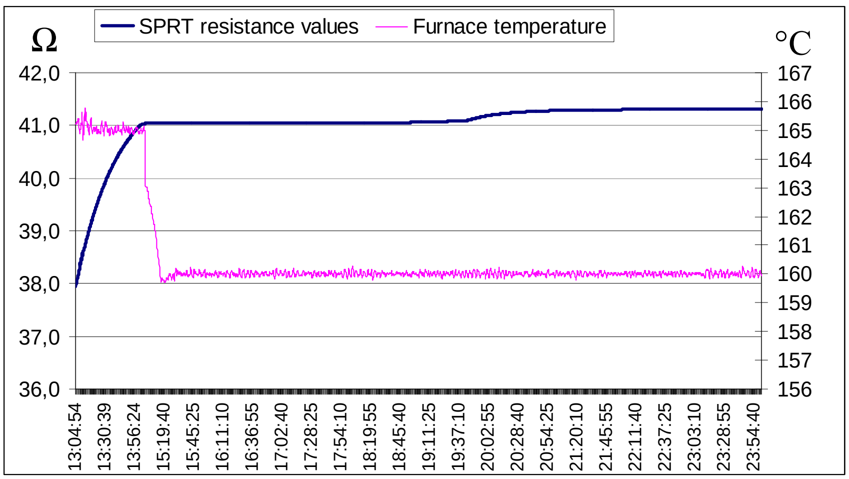

Fig. 4. The In melting process

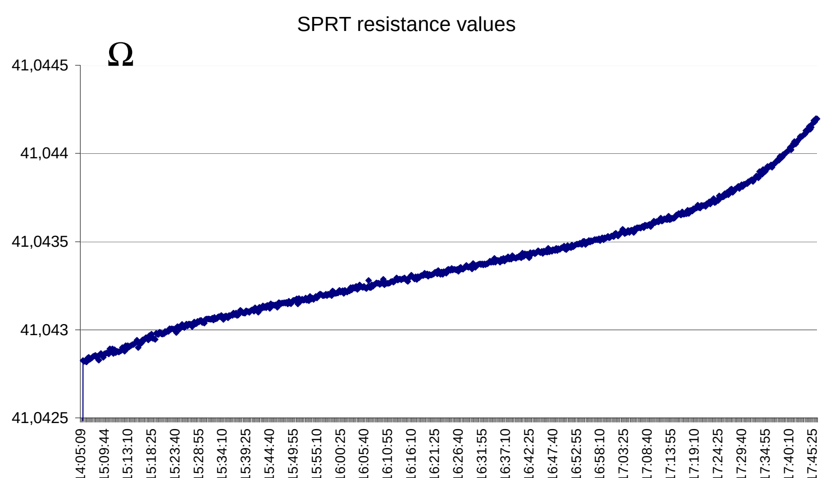

Fig. 5. The In melting process in detail

6. Indium freezing plateau realisation process measurement results

First the freezing process is analyzed. The following graphic illustrates data obtained for 16 hours. At the beginning of the process the furnace temperature is 160 °C (the pink chart – axis on the right). From this point the fixed point cell is cooling, so the SPRT (that is placed inside the cell) resistance values decrease (the blue chart – axis on the left). This trend stops when the SPRT resistance value is about 40.04 Ω.

At the beginning of the data acquisition period the furnace is set to 155 °C, so it needs about 20 minutes to cool (as seen on the pink chart in figure 6). After about one hour from the beginning of the data acquisition the indium forms the freezing plateau so the SPRT resistance (blue chart in Figure 6 – left axis) value stabilizes at 40.04 Ω for about 6 hours (figure 7 shows these values in detail). The furnace temperature is kept constant until the end of data acquisition.
Observing the chart in figure 7 the indium inside the fixed point cell formed a freezing plateau between 40.0420 Ω and 40.0435 Ω. If we take into consideration the values for 2 hours including the highest resistance values on the chart than the indium inside the fixed point cell formed a freezing plateau between 40.0434 Ω and 40.0435 Ω. Considering the fact that the SPRT is of type PT25, this resistance variation expressed in temperature measurement units is 0.001 °C. Figure 7 contains data from figure 6 (SPRT resistance value chart) showing only the freezing plateau.

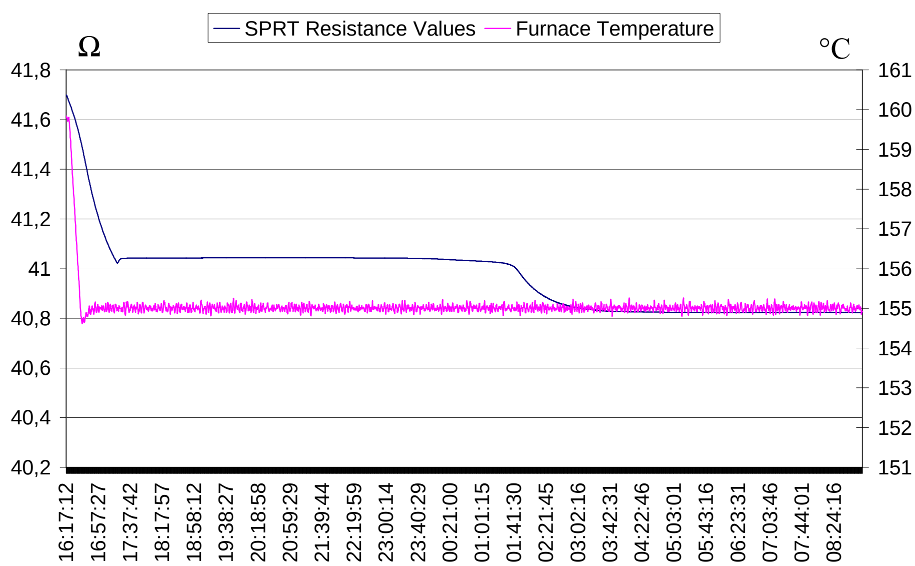

Fig. 6. The In freezing process

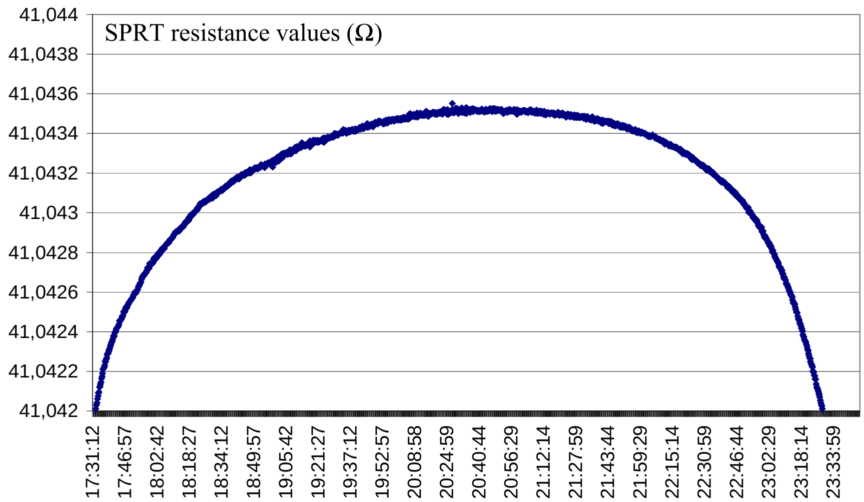

Fig. 7. The In freezing process in detail; SPRT resistance values

7. Conclusion

The software developed on Test Point platform offers some advantages for preset and future fixed point calibrations. The software brings some improvements because it makes the user's activity more efficient.

The first advantage is that the user can remotely set the furnace temperature values before and during the calibration process. Another advantage is the possibility to have real time data (calibration values: SPRT resistance values and furnace temperature) displayed on the PC monitor.

The main advantage is that the software saves the measured data for future analysis. This can lead to freezing plateaus with a better quality, this means plateaus that last longer, have a smaller temperature variation along the plateau duration and that are highly stable (don't freeze or melt locally).

From the values obtained from the freezing and melting plateaus some conclusions about the metal (in this case indium) purity can be obtained as well. These values are also useful to analyze the calibration furnace stability and behavior.

In the following studies an indium freezing plateau realization process automatization is prepared. In order to realize this a process optimization has to be made. That means to determine more accurately the moments when the software has to increase or decrease the furnace temperature, and its values.

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