BIPM Capacity Building & Knowledge Transfer Programme

2025 BIPM - TÜBİTAK UME Project Placement

REPORT

Project Name	Assuring the tracibility of SPRTs calibration within a national metrology laboratory, by using Fixed Points Method.
Description	The objective of this project is to establish the traceability chain to the ITS-90 in the temperature range from –40 °C to 420 °C. The first step involved the realization of fixed points using sealed cells of high-purity substances (Hg, H_2O, Ga, In, Sn, Zn), which, according to the International Temperature Scale of 1990, cover this range. This interval is primarily realized using Standard Platinum Resistance Thermometers (SPRTs), through interpolation between the defined fixed points and the prescribed interpolation equations. The SPRT is calibrated by measuring its resistance ratio at these reference points. Once calibrated, the SPRT can interpolate temperatures between the fixed points with millikelvin-level accuracy. These calibrated SPRTs will serve as national standards, against which the General Directorate of Metrology (DPM) will calibrate secondary standards and industrial thermometers.
Author, NMI	Manjola Doka, General Directorate of Metrology, Albania
Mentor at TÜBİTAK UME	Mr. Murat Kalemci, Temperature Laboratory, TÜBITAK-UME, Gebze/Kocaeli, Türkiye.
Date	September 1st, 2025 to October 3rd, 2025.

Motivation & Introduction

The Temperature Laboratory at DPM maintains six fixed points covering the ITS-90 measurement range from – 40 °C to 420 °C. These fixed points had never been used by the laboratory staff before. Participation in this training aimed to gain practical experience in realizing the fixed points and in calibrating SPRTs within this range.

Successful completion of the training will support us in achieving our key objectives:

1. Declaration of our first CMC in the field of thermometry.
2. Establishing internal traceability for the DPM laboratory, thereby reducing financial costs of obtaining traceability from other NMIs.
3. Ensuring traceability for the entire national economy.

Research

The training was structured into two phases:

- **Phase 1:** Realization of fixed points and execution of measurements for the calibration of SPRTs.
- **Phase 2:** Understanding and calculation of all contributors to the uncertainty budget in the calibration of SPRTs.

The *Guide to the Realization of the ITS-90 – Metal Fixed Points* describes essential details such as design, composition, and practical methods for realizing the fixed points of metals.

Before conducting primary-level calibrations, the Temperature Laboratory at DPM must be properly prepared. This means that, prior to starting the realization of fixed points, it is necessary to perform the characterization of all relevant instruments (such as furnaces and liquid baths), which are fundamental in this process.
The characterization of furnaces and baths is a critical step in the realization of fixed points, since the quality and stability of the thermal environment directly affect the reproducibility and accuracy of the fixed-point plateaus. The furnace or bath serves as the thermal envelope of the fixed-point cell. Characterizing it ensures that the cell operates under the correct, stable, and uniform conditions so that the true ITS-90 fixed-point temperature is realized with low uncertainty and high reproducibility. In essence, when we characterize a furnace or a bath for fixed-point realizations, we are measuring and documenting its behavior under controlled conditions. This process enables us to determine whether it provides a suitable thermal environment for the fixed-point cell.

Table 1: What we typically determine in the characterization process.

Parameter	How it is tested	Why it is important
Axial gradient	Place several thermometers/PRTs at different heights inside the cavity	Ensures that the entire fixed-point cell is within a uniform temperature zone
Radial gradient	Measurements at the center and near the cavity walls	Verifies that the center (where the cell is located) is at the same temperature as edges
Stability over time	Continuous monitoring of temperature for minutes/hours during a plateau	Good stability prolongs the plateau and keeps the temperature constant
Reproducibility	Repeat heating/cooling cycles at the same setpoint	Shows whether realization conditions can be reproduced with the same result
Heating and cooling	Evaluate ramping rates of the furnace/bath	Controlled rates prevent supercooling or incomplete melting
Heat flux / shielding	Compare measurements with and without thermal shields	Eliminates influence of radiation or convection that may distort the cell temperature
Plateau quality	Analyze duration and slope of the melting/freezing plateau	Long and flat plateau = accurate and stable realization of the fixed point

- **Realization of the Zinc Fixed Point**

1) Preparation of the furnace and the cell

Heat the furnace and the zinc cell to a temperature approximately *(Zn point + a few °C)*, typically about 5 °C above the zinc freezing temperature, and hold it to remove history effects. The SPRT must be preheated according to the laboratory's stabilization procedure. The furnace is usually heated overnight for 5–18 hours**.** Considering the furnace offset Δt, it is set to (420 °C±Δt) + t °C

2) Cooling to the zinc freezing plateau

In the morning, lower the temperature to 2 °C below 420 °C**,** which corresponds to the zinc freezing plateau. After 30–40 minutes of stabilization, initiate supercooling by inserting a rod into the cell for about 1 minute. In this way a thin mantle of metal freeze around the thermometer well. Withdraw the rod and place the SPRT into the cell. After ~15 minutes, start recording measurements.

3) Measurement sequence (0 °C – 420 °C, Zn)

Measure the SPRT resistance in the following order:

$R_{tpw} \rightarrow R_{Zn1} \rightarrow R_{tpw}$

(a)

(b)

(c)

(d)

Figure 1. Furnace for realizing the zinc freezing point (a) and the necessary accessories (b, c, and d)

- **Decision criterion:**

- If $\Delta R_{tpw} < 1$ mK → everything is acceptable, continue with the calibration procedure.
- If $\Delta R_{tpw} > 1$ mK → perform **annealing** of the SPRT for 2 hours at 450 °C.

Note: The annealing temperature is set +30 °C higher than the highest fixed point temperature at which the SPRT is being calibrated.

- **Repetition after annealing**

Repeat the sequence:

$R_{tpw} \rightarrow R_{Zn2} \rightarrow R_{tpw}$

Apply the same decision criteria:

- If $\Delta R_{tpw} < 1$ mK → proceed with SPRT calibration.
- If $\Delta R_{tpw} > 1$ mK → repeat the annealing process for another 2 hours at 450 °C.

4) Corrections applied

When determining the resistance value of the calibrated SPRT, two corrections must be applied are self-heating correction and hydrostatic head effect correction.

a) Self-heating correction

To determine the self-heating correction of the Unit Under Calibration (UUC), the resistance of the UUC is measured at the working current I=1mA, I√2mA and then again at I=1mA.

I = 1mA → R_1mA
I = √2 mA → $R_{\sqrt{2}}$ mA
I = 1mA → R_1mA

Equation (1) is the formula to calculate the self-heating correction.

$$R_0 = 2 * (\overline{R_{1mA}} - R_{\sqrt{2}mA}) \qquad (1)$$

b) Hydrostatic head correction

During measurement at a fixed point, the sensor of a SPRT is usually placed at a height which is "h" meters lower than the upper surface of the pure metal and where the pressure is higher than that at the surface due to the static head. ITS-90 gives all of the necessary coefficients for the calculation of the correction caused by the pressure difference, which are summarized in Table 2.

Table 2: Effect of pressure on the temperature of some defining fixed points

Substance	Assigned Value of Equilibrium Temperature T Kelvin (K)	Temperature with Pressure, p K1; dT/dp (10^{-8} mK/Pa)	Variation with Depth K2 : dT/dh (mK/m)	Approximate dW/dt
Argon (T)	83.8058	25	3.3	0.004342
Mercury (T)	234.3156	5.4	7.1	0.004037
Water (T)	273.16	–7.5	–.73	0.003989
Gallium (M)	302.9146	–2.0	–1.2	0.003952
Indium (F)	429.7485	4.9	3.3	0.003801
Tin (F)	505.078	3.3	2.2	0.003713
Zinc (F)	692.677	4.3	2.7	0.003495
Aluminum (F)	933.473	7.0	1.6	0.003205
Silver (F)	1234.93	6.0	5.4	0.002841
Gold (F)	1337.33	6.1	10	—
Copper (F)	1357.77	3.3	2.6	—
(T) – Triple Point (M) – Melting Point (F) – Freezing Point				

The correction of temperature caused by the difference in pressure can be calculated by using the equation (2):

$$\Delta t = h \times k_2 \qquad (2)$$

Where:
k_2 = dT/dh
h: the immersion depth of the midpoint of the sensor of a SPRT into the matter used for the fixed point.
From table 2, which is actually the same as table 2 of the ITS-90 document, for the freezing zinc point k_2 = 2.7 mK/m.

The following calculations were performed for the DPM zinc fixed point. The distance from the inner bottom of the central well to the surface of liquid metal is about 0.181 m. The SPRT sensor has a length of 5 cm. If the distance of the midpoint of the sensor from the tip of the sheath is 25 mm, the mean immersion depth of the SPRT sensor is: 181 mm – 25 mm = 156 mm = 0.156 m

The temperature correction, Δt, calculated using equation (2):
Δt = 0.156 m × 2.7 mK/ m

Consequently:
Δt =0.4212 mK = 0.0004212 K
ΔR = 0.0004212 mK /10000 Ω = 0.00004212 Ω

The real resistance value of SPRT (UUC) at TP Hg is then corrected as below:

$$R_{Zn} = R_{0SH} + \Delta R \qquad (3)$$

Where:
R_{0SH} - is the correction for self-heating
ΔR - is the correction for hydrostatic head effect
These two corrections are made for each fixed point at which the SPRT is calibrated.

Table 3: Hydrostatic head correction calculations for all fixed points of the DPM.

Substance	Equilibrium temperature T Kelvin (K)	Variation in relation to depth K2: dT/dh	Distance of the central well of the	Sensor midpoint distance (	Immersion Sensor depth SPRT (m)	Temperature correction, Δt (mK)	Correction (K)	Correction for SPRT (Ω)	Correction for Pt100 (Ω)	Calculation of the current temperature, t1=t+Δt (K)
Mercury	234.3156	7.1	0.182	25	0.157	1.1147	0.0011147	0.00011147	2.787E-05	234.3167147
Water	273.16	-7.3	0.270	25	0.245	-1.7885	-0.001117	-0.000112	-2.79E-05	273.1588831
Gallium	302.9146	-1.2	0.18	25	0.155	-0.186	-0.000186	-1.86E-05	-4.65E-06	302.914414
Indium	429.7485	3.3	0.182	25	0.157	0.5181	0.0005181	5.181E-05	1.295E-05	429.7490181
Tin	505.078	2.2	0.182	25	0.157	0.3454	0.0003454	3.454E-05	8.635E-06	505.0783454
Zinc	692.677	2.7	0.181	25	0.156	0.4212	0.0004212	4.212E-05	1.053E-05	692.6774212

➢ Realization of the Tin Fixed Point

1) Preparation of the furnace and the cell

Heat the furnace and the tin cell to a temperature approximately *(Sn point + a few °C)*, typically about 5 °C above the tin freezing temperature. The SPRT must be preheated according to the laboratory's stabilization procedure. The furnace is usually heated overnight for 5–18 hours**.** Considering the furnace offset Δt, it is set to (232 °C±Δt)+t °C

2) Cooling to the tin freezing plateau

In the morning, lower the temperature approximately to 227 °C for 2 hours**.** After 2 hours, the Sn cell is removed from the furnace and kept outside at ambient conditions (laboratory temperature) for 2 minutes and then placed back into the furnace. In this process, a thin metal shell forms around the cell. A rod at laboratory ambient temperature is then inserted into the cell's well for about one minute. This step promotes the formation of a uniform thin metallic layer surrounding the inner well of the cell. Withdraw the rod and place the SPRT into the cell. After ~ 15-30 minutes, start recording measurements. Two corrections, self-heating and hydrostatic head correction, are also applied to measurements in this cell.

Figure 2. Tin fixed-point cell in furnace with SPRT (left) and the tin fixed-point cell (right).

- **Realization of the Indium Fixed point**

The furnace is initially set a few degrees below the melting point of indium. The system is allowed to stabilize until both the cell and the bath reach thermal equilibrium, which typically requires 1.5 to 2 hours. After stabilization, the temperature is gradually increased until the indium melting point (156.6 °C) is reached. Melting starts at the outer wall of the cell and progresses toward the central channel where the SPRT is inserted. As the solid and liquid phases coexist, a melting plateau is established. Following the onset of melting, a waiting period of 30–45 minutes is recommended before starting accurate measurements with the SPRT.

- **Realization of the Gallium Fixed point**

The gallium fixed-point cell is placed in a heated isothermal enclosure. Since gallium melts just above room temperature, the realization of this fixed point is relatively simple. The system is allowed to stabilize thermally, and then the SPRT is inserted into the cell. Gallium begins to melt from the outer wall toward the central channel where the SPRT is located. When the solid and liquid phases coexist, the melting plateau is formed. Measurements with the SPRT are then carried out during the plateau. Some laboratories follow the practice of performing several melt–freeze cycles to prepare the cell before official measurements, as this increases uniformity.

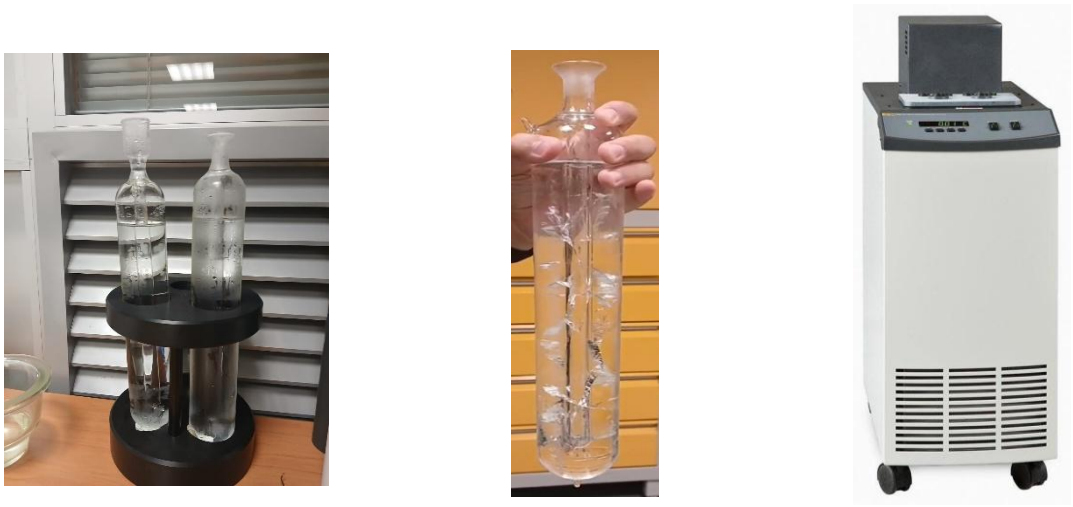
Figure 3. Gallium fixed-point cell in a heated isothermal enclosure

- **Preparation and Maintenance of the Triple Point of Water Cell**

One day prior to use, the cell is placed in a water bath stabilized at 0.01 °C, which serves for its maintenance. This step allows the cell to acclimatize to the environmental conditions under which the realization will be performed. After 24 hours, the cell is removed from the bath.
For the realization process, crushed dry ice is required. Initially, the cell is cleaned by introducing pure alcohol into the inner well, gently swirling it, and carefully pouring it out. This procedure is repeated three times to ensure that the well is completely free of contaminants.
Subsequently, crushed dry ice is introduced into the well. The cell is then tapped lightly with the palm of the hand. A rod is not used to push the dry ice further down, as this would require holding and warming the lower part of the cell by hand. This can cause significant discomfort due to cold exposure and may pose health risks to the operator. The chosen method, although slower and more time-consuming, is safer both for the operator and for the integrity of the fixed-point cell.

After approximately 30 minutes, ice formation begins within the cell. The remaining dry ice is carefully leaving to the well, and a characteristic cracking sound (“crack”) indicates the formation of ice inside the sealed water cell. Immediately afterwards, the cell is placed back into the water bath at 0.01 °C and left overnight for stabilization.

On the following day, the cell is inspected. By this stage, the internal ice structure has become more compact and homogeneous. The cell is then removed from the bath, and the inner well is rinsed at least three times with cold water from the ice point. This step facilitates the detachment and redistribution of the ice formed around the inner well.
Finally, the inner well is filled with distilled water mixed with a few drops of alcohol, maintle ice should and the cell is returned to the maintenance bath at 0.01 °C, ready for use for calibration of SPRT.



(a) (b) (c)

Figure 4. (a) Water triple-point cell before realization, (b) realized cell with dry ice, (c) Maintenance bath for water triple-point cell: before and after realization

- **Realization of the Mercury Triple Point**

The mercury fixed-point cell is placed in the central position of the bath. A special holder is used to ensure full immersion and mechanical stability of the cell. The SPRT is inserted to the required depth and properly secured. The bath is switched on and set to a temperature about 5–10 °C below the melting point of mercury ~ 45 °C. This ensures that the entire mercury mass inside the cell solidifies. This stage typically requires 1.5–2 hours for complete stabilization of both the bath and the cell. Once the cell is fully solidified, the bath temperature is slowly increased until it approaches the melting point (−38.8 °C). At this stage, the mercury begins to melt from the outside toward the inner channel surrounding the SPRT.

The melting plateau then starts to form. After 30–45 minuta measurements with SPRT are perfomed.

Figure 5. Bath for realizing the mercury triple point with the mercury cell

- **Calibration Procedure**

For the calibration of a SPRT between -40°C and 420°C, the sequence (after stabilized) would be:

- Measurement at Ga M.P.
- Measurement at WTP
- Measurement at Zn F.P
- Measurement at WTP
- Measurement at Sn F.P
- Measurement at WTP
- Measurement at Hg T.P
- Measurement at WTP

- **Uncertainty budget**

The main contributions can be divided into 4 groups:

- Fixed-point effects: hydrostatic pressure, residual gas pressure, impurity and isotopic effects and thermal effects
- SPRT effects: oxidation, impurities, insulation and leakage
- Interpolation effects: non-uniqueness
- Resistance measurement effects: reference resistor, connecting cables, resistance bridge effects, and self-heating

For each uncertainty component, a standard uncertainty u_i and its associated degrees of freedom must be provided. The value of u_i should be given in terms of temperature. For type A evaluation, the number of degrees of freedom, is n-1 where n is the number of measurements.

For type B evaluation, any input is assumed to have an infinite number of degrees of freedom. The combined uncertainty U, the effective degrees of freedom and, subsequently, the expanded uncertainty at 95% level of confidence are calculated as set out in the Guide.

W_t is determined according the following mathematical model obtained from the relationship

$$W_t = \frac{(R_S+C_{Rs/3}+C_{Rs/4})*(X_t+C_{Xt/1}+C_{Xt/2}+C_{Xt/3}+C_{Xt/4}+C_{Xt/5}+C_{Xt/6})}{(R_S)*(X_{0.01°C}+C_{x0.01/1}+C_{x0.01/2}+C_{x0.01/3}+C_{x0.01/4}+C_{x0.01/5}+C_{x0.01/6})} \quad (4)$$

$$W_t = (1 + D_{Rs/3} + D_{Rs/4}) * \frac{(X_t+C_{Xt/1}+C_{Xt/2}+C_{Xt/3}+C_{Xt/4}+C_{Xt/5}+C_{Xt/6})}{(X_{0.01°C}+C_{x0.01/1}+C_{x0.01/2}+C_{x0.01/3}+C_{x0.01/4}+C_{x0.01/5}+C_{x0.01/6})} \quad (5)$$

Where:

Rs: reference resistor value at the time of TPW measurement
$D_{Rs/3}$: relative drift of the resistance of the reference between TPW and FP measurements
$D_{Rs/3} = C_{Rs/3} / Rs$ (Negligible if measurement performedin a short time)
$D_{Rs/4}$: relative temperature variation of resistance of the reference between TPW and FP measurements, $D_{Rs/4} = C_{Rs/4} / Rs$

Effects linked with triple point of water calibration:

$X_{0.01°C}$	reading on the bridge at the TPW (same SPRT, same cell, same day)
$C_{x0.01/1}$	water triple point reference including isotope variation
$C_{x0.01/2}$	Hydrostatic pressure correction (estimated from the uncertainty of the sensible element position and the uncertainty of the free liquid level)
$C_{x0.01/3}$	Perturbing heat exchanges (deviation from expected hydrostatic pressure correction obtained by changing immersion depth)
$C_{x0.01/4}$	Self-heating correction (resolution of the bridge readings, uncertainty on the ratio between the two measuring currents)
$C_{x0.01/5}$	Bridge linearity (use of calibrated resistor for checking the bridge)
$C_{x0.01/6}$	SPRT internal insulation leakage correction (Decrease in resistance over some hours in the triple point)

Effects linked with the considered fixed point calibration:

X_t	Reading on the bridge (same SPRT, same cell, same freezing, same day)
$C_{Xt/1}$	Chemical impurities (sum of individual estimates)
$C_{Xt/2}$	Hydrostatic pressure correction (estimated from the uncertainty of the sensible element position and the uncertainty of the free liquid level)

$C_{Xt/3}$	Perturbing heat exchanges (deviation from expected hydrostatic pressure correction obtained by changing immersion depth)
$C_{Xt/4}$	Self-heating correction (resolution of the bridge readings, uncertainty on the ratio between the two measuring currents)
$C_{Xt/5}$	Bridge measurement correction, lack of linearity (use of calibrated resistor for checking the bridge)
$C_{Xt/6}$	Gas pressure correction (sealed cells: uncertainty on pressure measurement during sealing combined with temperature profile)

Table 4: Uncertainty from impurities, typically used in calibration budget when no chemical analysis is available

Fix point (ITS-90)	Temperature	Uncertainty from impurities
Hg triple point	–38.8344 °C	≈ 0.3 mK
Water triple point	0.01 °C	≈ 0.05 mK (from impurities + isotopes)
Ga melting point	29.7646 °C	≈ 0.05 mK
In freezing point	156.5985 °C	≈ 0.3 mK
Sn freezing point	231.928 °C	≈ 0.5 mK
Zn freezing point	419.527 °C	≈ 0.7 mK

Conclusions and Future Work

An interesting fact was that most of the equipment used for the realization of fixed points here in the primary temperature laboratory at TÜBİTAK-UME was the same as the equipment already available in the Temperature Laboratory at DPM. This gave me both confidence and assurance that the knowledge gained during the training would not only be valuable, but also much easier to apply in practice.

The Temperature Laboratory at DPM currently lacks some necessary accessories, such as the glass wool and the proper holder for the mercury triple-point cell within the bath. The laboratory staff constructed an improvised holder using a plastic tube and available materials. The mentor for this training, Mr. Murat Kalemci, confirmed that this solution is acceptable and will not cause any issues in realizing the mercury fixed point.

(a) (b)

Figure 6: Holder for the mercury triple-point cell within the bath (a), and glass wool (b)

During 2026, with the support of the laboratory of temperature here at Tubitak-UME, we aim to carry out:

- Supplementary key comparison on PRT calibration from -50°C up to 280°C
- Bilateral Key Comparison on SPRT calibration from the triple point of Hg to the Freezing point of Zn

This would help us achieve the objectives of this project, which are:

1. The declaration of our first CMC in the field of thermometry.
2. Establishing in-house traceability for the DPM laboratory, thereby reducing the financial costs associated with obtaining traceability from another NMI.
3. Ensuring traceability for the entire economy of our country.

Acknowledgements

During my stay here, I truly felt at home. My heartfelt thanks go to the entire laboratory staff, especially Mr. Murat Kalemci and Ms. Narcisa Arifovic, whose contributions I am certain will greatly support me in achieving the objectives of this project. I am very pleased that they were willing to assist the Temperature Laboratory at DPM in carrying out a supplementary comparison for the calibration of platinum resistance thermometers (PRTs).

I would also like to sincerely express my appreciation to Dr. Enver Sadıkoğlu and Ms. Müge Atam from the International Relations Department at TÜBİTAK UME, as well as Mr. Chingis Kuanbayev and Mr. Anderson Maina from the BIPM Capacity Building and Knowledge Transfer management, for organizing these project placements and ensuring excellent accommodation during the program.

My sincere gratitude also goes to the leadership of the General Directorate of Metrology (DPM) for their support and trust in both me personally and in my work. I hope that next year one of my colleagues at DPM will also be selected and have the opportunity to benefit from this wonderful experience at UME.

My deepest thanks also go to my family, and especially to my husband, for his unconditional support.

Finally, I would like to thank all participants of the 8th cycle of the BIPM–TÜBİTAK UME program, who made my days in Turkey more beautiful and unforgettable. I will cherish our wonderful memories and look forward to future collaborations between our institutions.

Bibliography

[1] The International Temperature Scale of 1990, H.Preston Thomas, Metrologia, 27, 3-10

[2] Guide to the Realization of ITS-90, Platinum Resistance Thermometry

[3] Guide to the Realization of ITS-90, Triple Point of Water

[4] Guide to the Realization of ITS-90, Metal Fixed Points for Contact Thermometry

[5] Guide to the Realization of ITS-90, Fixed Points: Influence of Impurities

[6] Methods of reducing the uncertainty of the self-heating correction of a standard platinum resistance thermometer in temperature measurements of the highest accuracy, V.Batagelj, J.Bojkovski and J.Drnovˇsek