XIX IMEKO World Congress
Fundamental and Applied Metrology
September 6–11, 2009, Lisbon, Portugal

PRACTICAL LIMITS OF MEASUREMENT UNCERTAINTIES IN CALIBRATION OF STANDARD PLATINUM RESISTANCE THERMOMETERS BY COMPARISON

Jovan Bojkovski [1], Valentin Batagelj [1], Janko Drnovšek [1], Vincencij Žužek [1]

[1] University of Ljubljana, Faculty of Electrical Engineering, Laboratory of Metrology and Quality, Trzaska 25, SI-Ljubljana, Slovenia, jovan.bojkovski@fe.uni-lj.si; valentin.batagelj@fe.uni-lj.si; janko.drnovsek@fe.uni-lj.si

Abstract – Usually, SPRTs are calibrated at fixed points, but due to the high cost of a primary realization of physical quantities and time needed for the calibration at fixed points, it can be reasonable to calibrate them by comparison at the highest level.

The calibration by comparison is a technique most widely used to calibrate measuring instruments, not only in industry but also in many secondary calibration laboratories. Calibration procedures of a typical secondary laboratory are based on the use of transfer standards, which are usually calibrated in a primary laboratory, thus providing traceability to (inter)national standards through a process of dissemination of a unit with an associated uncertainty.

The aim of the paper is to show that temperature calibrations by comparison, in the range from –95 °C up to 300 °C, can result in a calibration uncertainty (k=2) of 0,005 °C, which is sufficiently low for many industrial applications and secondary calibration laboratories.

Keywords: calibration by comparison, standard platinum resistance thermometer, uncertainty

1. INTRODUCTION

In order to make optimal use of Standard Platinum Resistance Thermometers (SPRTs) are calibrated at fixed points, but due to the high cost and time required for calibration at fixed point, the laboratory can also decide to calibrate SPRT by comparison with an uncertainty which can be sufficiently close, for many applications, to the uncertainty achieved by calibration at fixed points. Some calibration laboratories are also now using slim fixed point cells operated in block calibrators, with the argument that such a calibration is more precise than a calibration by comparison, but takes less time. The calibration by comparison, in this case, is a technique when you associate a value of the temperature, as measured with a ITS-90 calibrated reference SPRT thermometer, with the resistance value of the SPRT under test, inside stable and homogeneous thermostatic baths/furnaces. This procedure is repeated at different temperatures within the range of the SPRT under test and the appropriate interpolation equation is calculated.

In the calibration of resistance thermometers by comparison, several uncertainty contributions have to be taken into account. However, there are practical and theoretical constraints in achieving the same ultimate limits in uncertainty of calibration of thermometers by comparison as are achievable with fixed point calibration. A number of uncertainty contributions can be further reduced without any significant influence to the total uncertainty. As an example, we can use better bridge together with better standard resistor, but total uncertainty will not be significantly decreased.

In this article uncertainty sources in the range from –95 °C up to 300 °C will be analyzed in details. This is the range in which laboratory can use classical calibration baths with various liquids as calibration media. The paper will show that, in this range, the uncertainties achieved in calibration by comparison can be sufficiently close, for many industrial applications, to the uncertainties achieved by calibration at fixed points.

2. THE UNCERTAINTY SOURCES IN CALIBRATION OF THE SPRT BY COMPARISON

In general, there are eight typical uncertainty sources that we can identify in process of calibration of the SPRT by comparison. Only two are type A uncertainty and all others are type B uncertainty. These contributions are:

- the standard deviation of the readings (type A), which also represents bath stability during the particular calibration point;
- the uncertainty of the reference thermometers used in process of the calibration;
- the contribution of the thermostatic bath (axial and radial gradient - uniformity) used as a calibration medium;
- the repeatability obtained from the fit residuals of unit under calibration (type A);
- the uncertainty of the resistance bridge and its resolution;
- the uncertainty of the standard resistors together with the stability of the standard resistor thermostat;
- the uncertainty due to the immersion error
- the uncertainty due to the self-heating,

as described in [1], [2] and [3]. Some of these uncertainty sources can be easily determined from

calibration certificates of the equipment used, but some have to be measured and/or estimated.

Since we are calibrating by comparison, the first contribution, which we cannot avoid, is the uncertainty of the reference thermometer. The reference thermometer used in calibration by comparison at the highest level is typically standard platinum thermometer (SPRT) calibrated at fixed points. The expanded uncertainty, together with its stability, of the SPRT calibrated from the triple point of argon (-189,3442 °C) up to the freezing point of zinc (419,5270 °C), which can be routinely achieved in the national laboratories, is 1 mK (k = 2).

A calibration bath cannot be considered as completely stable in time and homogeneous all over its volume, especially when temperature calibrations by comparison are performed at the best level of uncertainty. This represents a major contribution to the total uncertainty of a calibration procedure.

In order to decrease this uncertainty contribution, we are using equalizing blocks in all our calibration baths. The dimension of the block depends on the bath dimension. The height of the block is at least 100 mm, so that sufficient immersion into the block can be achieved. The wells in the block have different diameters, so that thermometers with different diameters can be calibrated at the same time, as well as to allow better thermal conductivity between block and the thermometer, as described in [4].

In the range from –95 °C up to 300 °C, we are using four different baths. The range between –95 °C and 10 °C is covered by the methanol cryostat bath, the range between 10 °C and 85 °C by the water bath, the range between 85 °C and 150 °C by a light viscosity oil bath and the range between 150 °C and 300 °C by a high viscosity oil bath. The characteristics of each bath are presented in the Table 1. All the uncertainty contributions in table were measured at the laboratory, are expanded for k=2 and represent the largest contribution in the relevant range.

Table 1. The characteristics of the baths used for the calibrations by comparison (with equalising block)

Range	Media	Supplier	Uniformity	Stability
-95 °C to 10 °C	Methanol	Hart	2,5 mK	1 mK
10 °C to 85 °C	Water	Kambič	2 mK	1 mK
85 °C to 150 °C	Light viscosity oil	Kambič	2 mK	1 mK
150 °C to 300 °C	High viscosity oil	Kambič	2 mK	1,5 mK

The relationship between the resistance of the platinum resistance thermometer under calibration and temperature measured with a reference thermometer is described with an interpolation equation. This equation is determined using the least-squares method on the data acquired during the comparison. There are several choices of interpolation equation.

In this paper we will present three different equations, the Callendar-van Dusen (CVD) equation, [5], as presented with the following equation:

$$R_t = R_0 \cdot \left(1 + A \cdot t + B \cdot t^2 + C \cdot \left(t - 100\ ^{\circ}\mathrm{C}\right) t^3\right) \quad (1)$$

where $C = 0$ for $t > 0$ °C,

the W-W_r equation, as presented in the following equation,

$$W(T_{90}) - W_r(T_{90}) = \Delta W_i(W_r(T_{90})) \quad (2)$$

where ΔW_i are deviation functions from the W_r (reference function defined in ITS-90), chosen on the basis of the range, as described in details in [6], and ordinary polynomial fit of n-th order (in our case fifth order), as presented with the following equation,

$$R_t = R_0 \cdot \left(\sum_{i=0}^{n} a_i \cdot t^i\right) \quad (3)$$

The CVD equation is generally accepted as interpolation equation for industrial platinum resistance thermometers (IPRTs), rather than for the SPRTs. While, the ITS-90 equation is usually used for the presenting of the results of the SPRTs calibrated at fixed points. The ordinary polynomial fit of higher order (fifth and higher) is derived from the CVD equation.

For the purpose of this paper, we calibrated three different thermometers: 25 Ω SPRT Rosemount with the metal sheath; 25 Ω SPRT Tinsley with the quartz sheath and 100 Ω PRT Isotech with the quartz sheath.

As a reference we used another 25 Ω SPRT calibrated at fixed points. The measurements were performed in the range from –95 °C up to 300 °C at –95 °C, -90 °C and then in steps of 10 °C until 300 °C. That means for each thermometer we had 41 different measured points with repeated measurement in the ice point at the end of the calibration. The value of the resistance at measurements of ice point was averaged and used once in process of fitting. The repeatability of resistance converted to temperature at ice-point was well within 0,5 mK.

The residual errors, for fits where all 41 measured points are used, are presented on the figures 1,2 and 3. The standard deviation of the residual errors in the fit, taking into account degrees of freedom, is presented in the Table 2. In this table all measured values were used for the determination of the equation and standard deviation.

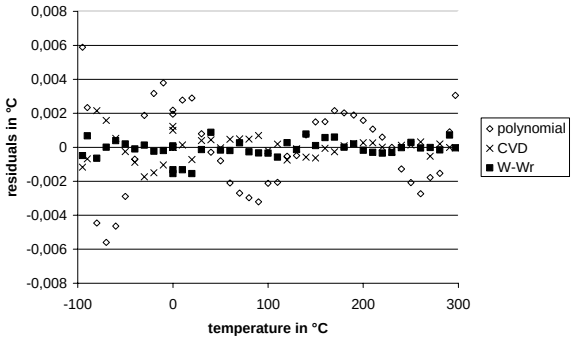

Fig. 1. The residual errors for Tinsley 25 Ω thermometer (5th order polynomial function)

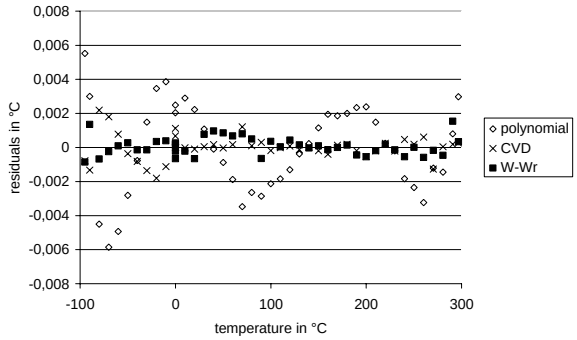

Fig. 2. The residual errors for Rosemount 25 Ω thermometer (5th order polynomial function)

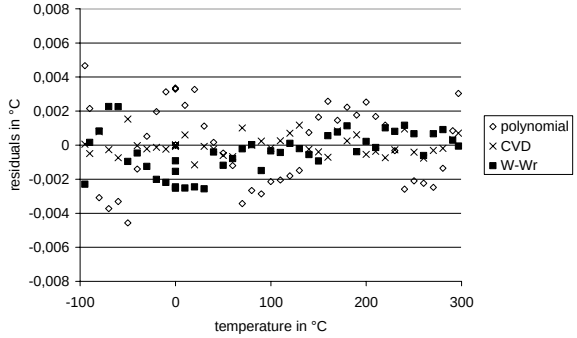

Fig. 3. The residual errors for Isotech 100 Ω thermometer (5th order polynomial function)

Table 2. The standard deviation of the residual errors in the fit

Quantity	Tinsley 25 Ω	Rosemount 25 Ω	Isotech 100 Ω
s(W-Wr)	0,6 mK	0,6 mK	1,5 mK
s(CVD)	0,8 mK	0,8 mK	0,7 mK
s(poly. 5th)	2,8 mK	2,9 mK	2,7 mK

It required 10 working days for the calibration at all 41 temperature points. Since the idea of the paper is also to decrease the time of the calibration of the SPRT with respect to the fixed point calibration (20-25 days), while keeping an acceptable expanded uncertainty (k=2) of < 5 mK, we tried the effect of decreasing the number of the temperature points required for the calibration. For the sake of the experiment, we tried to fit all three equations with 10 measured points and finally with 6 measured points, equally spacing the measured points over the range of the calibration. The residual errors for Tinsley 25 Ω thermometer, for fits where 10 measured points and 6 measured points are used, are presented on the figures 4 and 5. The other two thermometers exhibited similar behaviour.

The standard deviation of the residual errors increased from $s(W\text{-}W_r)$ = 0,6 mK for 41 measured points to $s(W\text{-}W_r)$ = 1,1 mK (10 measured points) and $s(W\text{-}W_r)$ = 1,2 mK (6 measured points).

Due to small degrees of freedom, we can conclude that calibration of the range from –95 °C to 300 °C, requires at least 10 equally dispersed points over the range. These 10 measurements can be made over 3 days, which is far less time than typical calibration at fixed point, in this range.

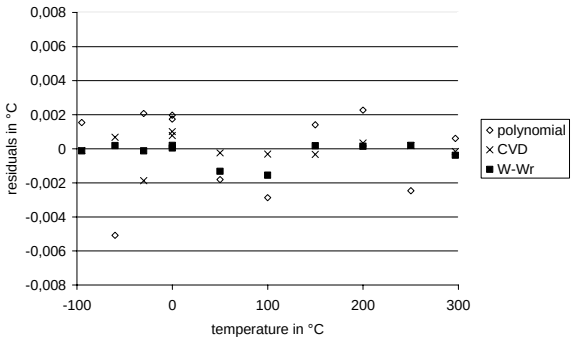

Fig. 4. The residual errors for Tinsley 25 Ω thermometer (10 measuring points) (5th order polynomial function)

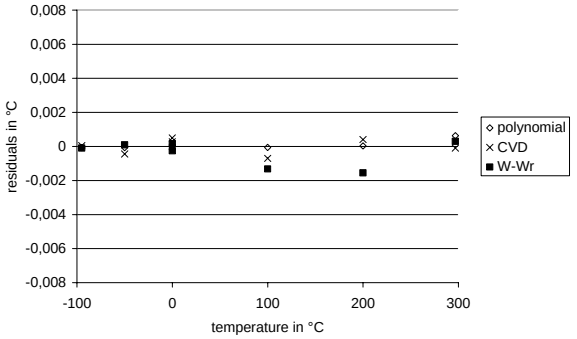

Fig. 5. The residual errors for Tinsley 25 Ω thermometer (6 measuring points) (5th order polynomial function)

The standard deviation of the readings of both thermometers, the reference one and the calibrated one, is directly correlated with the stability of the bath and short term stability of the reference resistor bath and resistance bridge. This uncertainty component was determined as the standard deviation of the readings and is bath dependent.

In our experiment, we used AC bridge ASL F 700 B, which is typically used in the secondary laboratory in the process of the calibration by comparison. The bridge uncertainty together with the resolution of the bridge is 0,5 ppm of measured value (i.e. for 25 Ω SPRT at 300 °C, the uncertainty 0,5 ppm is 0,275 mK).

The standard resistor used for the measurement was 25 Ω or 100 Ω depending on the thermometer measured. They were calibrated by our national electrical laboratory and the uncertainties of the resistors are 0,7 ppm. Of course, this value is even smaller if we report only *W* value and not resistance. In that case, only short term stability of the reference resistor during measurement is critical.

The immersion error was estimated by measuring the thermometer at different immersion depths. There were no detectable changes (less than 0,25 mK).

When SPRT resistance is being measured, measurement current will cause heat dissipation and increase the temperature of the SPRT sensor. Therefore it should be corrected, leaving only a relatively small uncertainty of this

correction, as explained in details in [7]. The uncertainty of the measurement of self-heating was estimated to be 0,2 mK.

3. DIFFERENCE BETWEEN CALIBRATION BY COMPARISON AND FIXED POINT CALIBRATION

In order to properly evaluate calibration by comparison, all three thermometers were also calibrated at fixed point. The differences between a calibration at fixed points and a calibration by comparison for Tinsley 25 Ω are presented in the figures 6, 7 and 8.

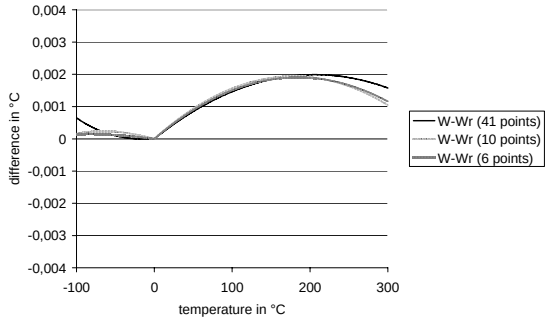

Fig. 6. The difference between W-W_r equation fitted using 41,10 or 6 points and W-Wr from the calibration at fixed points for Tinsley 25 Ω thermometer

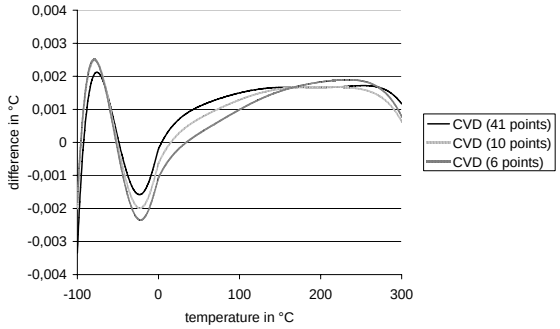

Fig. 7. The difference between CVD equation fitted using 41,10 or 6 points and W-W_r from the calibration at fixed points for Tinsley 25 Ω thermometer

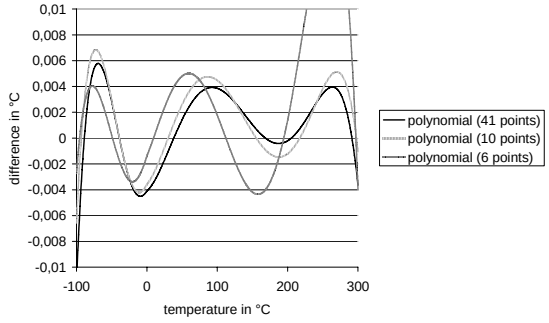

Fig. 8. The difference between polynomial fit of fifth order fitted using 41,10 or 6 points and W-W_r from the calibration at fixed points for Tinsley 25 Ω thermometer

Other thermometers show similar behaviour.

As one can see from these differences, ordinary polynomial fit is completely unacceptable for top level calibration of SPRT by comparison. The most appropriate is, as expected, determination of the (*W-Wr*) equation on the basis of the measurement by comparison and measurement at the triple point of water. Also acceptable for routine calibration is CVD equation.

6. CONCLUSIONS

As shown in this paper, with the analysis of all the relevant uncertainty sources and the state of the art equipment, it is possible to achieve the uncertainties in the calibration by comparison of the SPRTs which are sufficiently close for many industrial applications to the calibrations at fixed points in certain subranges. In this relatively limited range between –95 °C and 300 °C, the uncertainty of the 5 mK, for the calibration of the SPRTs is achievable, using state of the art equipment and reference standard. Some of the uncertainties can be further decreased, like uncertainty of the resistance bridge, but it is a question if it is worthwhile investigating and investing additional resources in decreasing these uncertainties as well as equipping a fixed point calibration laboratory. It is possible to achieve satisfactory uncertainty even with 10 calibration points, which can be measured in only 3 days.

The cost of the calibration by comparison is at least two times lower, if compared to the calibration at the fixed points. However, traceability of such calibration is not directly to the ITS-90 defining fixed points, but depends on the reference thermometer, which has to be calibrated at fixed points by a primary laboratory.

REFERENCES

[1] Drnovšek, J., Pušnik, I., Bojkovski, J., Meas. sci. technol., vol. 9, no. 11, pp. 1907-1911 (November 1998).

[2] Dubbeldam, J., de Groot, M., "Uncertainty Budget For Calibrations of Industrial PRTs" presented at the EUROMET workshop in Berlin, February 2001.

[3] Bojkovski, J., Pušnik, I., Drnovšek, J., "Uncertainty budget in calibration of the resistance thermometers by comparison" in Quality Manual of the MIRS/FE-LMK, , Ljubljana: MIRS/FE-LMK, 1997, pp. 14-16.

[4] Drnovšek, J., Bojkovski, J., Pušnik, I., Instrum. sci. technolog., vol 26, no. 5, pp 491-501 (December 1998)

[5] Nicholas, J. V., White, D., R., Traceable Temperatures, Chichester: John Wiley & Sons Ltd, 1994, pp. 153-198

[6] Preston-Thomas H., Metrologia, 27, pp 3-10 (1990)

[7] Batagelj, V., Bojkovski, J., Drnovšek, J., Pušnik, I., "Influence Of SPRT Self-Heat On Measurement Uncertainty In Fixed Point Calibration And Calibration By Comparison" submitted for the Temeprature 2002 Symposyum, Chicago: American Institute of Physics, 2002

[8] Batagelj V., Bojkovski J, Drnovšek J., Meas. sci. technol., 2003, vol. 14, pp. 2151-2158