The Development of a High-Temperature PRT Calibration Process Based On Dry-Block Calibrators

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Abstract: Industry trends are leading toward the need for improved accuracy in field calibration applications. The dry-block calibrator is the most common type of heat source used in field temperature calibrations but can be difficult to correctly address in a comprehensive uncertainty analysis. Significant sources of error or uncertainty are often overlooked or underestimated.

It is proposed that through the use of modern best practices in temperature metrology, the sources of error can be properly accounted for and managed to result in calibration uncertainties from 20 to 50 mK (milliKelvin) over the range -40 to 660 °C in a dry-block calibrator based PRT (Platinum Resistance Thermometer) calibration system. This will be demonstrated through the presentation of a system recently designed for a commercial secondary-level calibration laboratory. Included in the paper are a comprehensive uncertainty analysis as well as the problems, solutions, and results encountered during the design of the process.

The intent of this paper is to help calibration professionals in industry recognize and properly deal with sources of uncertainty related to dry-block type calibrators in temperature calibration processes. With modern best practices in temperature metrology it is possible to achieve reliable high-accuracy temperature calibrations, even in the field.

Key words: PRT calibration, field calibration, uncertainty analysis

1. INTRODUCTION

The Fluke Corporation, Hart Scientific Division calibration laboratory was asked to create a calibration solution for a new -200 to 660 °C, metal sheath PRT. The calibration system had to accommodate 6.35 mm (0.25 inch) diameter, straight sheath probes as well as 6.35 mm (0.25 inch) diameter, probes with a 90° bend, 241.3 mm (9.5 inches) from the tip (Figure 1). The calibration uncertainty needed to be ±50 mK or less over the entire calibration range.

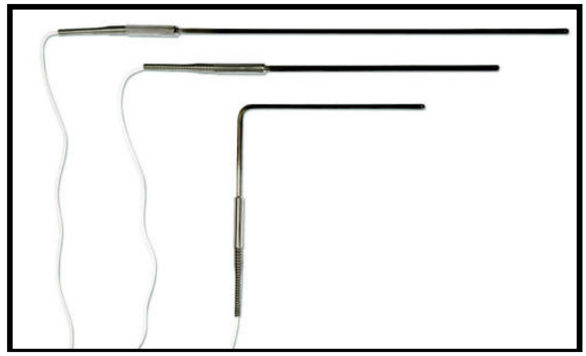

Fig. 1. Bent-sheath and Straight-sheath PRT Thermometers

After reviewing the options, the cal lab team decided to calibrate the new probes by comparison with a metal sheath SPRT (Standard Platinum Resistance Thermometer) in dry-block calibrators and a liquid-nitrogen comparator. The dry-block calibrators would be used over the range -40 to 660 °C and the liquid-nitrogen comparator would be used at -197 °C.

2. DESIGN CONSIDERATIONS

The cal lab's existing PRT calibration processes based on stirred-liquid baths worked very well but they were not able to provide temperatures above 500 °C. Also, they could not accommodate 90° bent-sheath probes due to the thickness of the bath lids. Other important considerations for the new process were the need to automate the measurements as much as possible and to provide sufficient process capacity to meet expected demand.

The combination of dry-blocks and liquid-nitrogen comparator were found to be the best option to cover the required temperature range while also accommodating the bent-sheath probes. The dry-block calibrators were equipped with RS232 communication to provide automation capability but calibration capacity was a concern due to the relatively small measurement inserts in the dry-blocks (Figure 2).

In order to achieve good temperature uniformity, the dry-blocks were designed with smaller inserts. To allow spacing for the handles of straight probes, the inserts could only be drilled with four holes (Figure 2). The team decided to add a fifth hole in the center of the insert for the reference SPRT. The additional hole increased capacity from 2 UUTs to 3.

The center hole was drilled to a diameter of 5.72 mm for the 5.56 mm diameter SPRT while the surrounding holes used for UUT measurement were drilled to a diameter of 6.63 mm for the 6.35 mm (0.25 inch) diameter UUTs. In the hot dry-block (9173) the holes were drilled to a depth of 203 mm while the hole-depth in the cold dry-block (9170) was drilled to 160 mm.

Probe Model	Probe Diameter	Sensor Length	Sheath Material
5699 SPRT	5.56 mm	30 mm	Inconel™
5609 PRT	6.35 mm	45 mm	Inconel™

Table 1. Probe Dimensions and Sheath Material

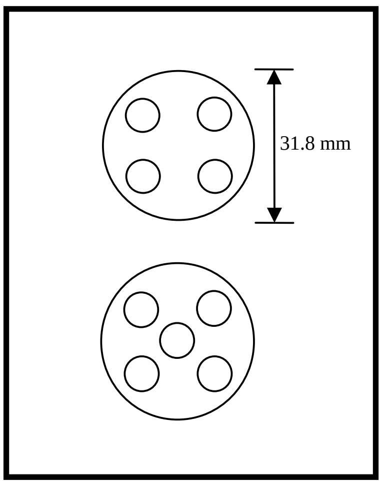

Fig. 2. Dry-Block Insert Hole Configurations

Typically, dry-block calibrator inserts are designed so the center of each measurement well is on the same radius around the insert with the holes evenly distributed to maintain symmetry and equidistance from the heaters located around the insert (as top half of Fig. 2). The risk with using a hole in the center is that the hole-to-hole (radial) gradients could be worsened since the center hole is not the same distance from the heaters as the other holes. However, in this application, this is not an issue since the measurements are a comparison between a reference SPRT (center hole) and each UUT (outer holes). To be sure, both designs were tested and proven to meet the gradient specifications in the uncertainty analysis. The team chose to use the 5-hole insert to increase capacity by one. A capacity of three UUTs was found to be sufficient for process demand.

3. CALIBRATION PROCESS

The UUTs are calibrated by comparison with a reference SPRT using the dry-block and liquid nitrogen calibrators as heat sources. The calibration points and the order they are taken in are listed in Table 2. The RTPW (Resistance at the Triple-Point of Water) is measured multiple times throughout the calibration to monitor the short-term repeatability of the UUT. The RTPW measurements are performed by comparison in the dry-block calibrator as well.

Step	Set Point (°C)	Heat Source
1	0.01 (As Found RTPW)	9170
2	0.01 (Begin RTPW)	9170
3	157	9173
4	232	9173
5	420	9173
6	660	9173
7	232 (Hysteresis Check)	9173
8	0.01 (Mid RTPW)	9170
9	-39	9170
10	-197.0	Liquid Nitrogen
11	0.01 (Final RTPW)	9170

Table 2. Calibration Set Points

After the As Found RTPW point is taken, the probes are soaked at 665 °C for 120 minutes to pre-treat them before calibration.

At each temperature the dry-block is allowed to stabilize for a defined period of time to allow optimum temperature uniformity in the insert before data is taken. Once data is taken it is analyzed for stability before the data is accepted.

After the data is taken, the calibration temperature vs. resistance data are fit to the ITS-90 using the deviation function sub ranges 4 (-189.3 to 0.0 °C) and 7 (0.0 to 660.3 °C).

4. CALIBRATION QUALITY CONTROL

Several checks are implemented in the calibration process to ensure quality. As previously mentioned, at each set point the measurement standard deviation is monitored per limits established in the uncertainty analysis. As previously mentioned, the RTPW measurements are used to evaluate the short-term repeatability of each UUT. The RTPW short-term allowance is also established in the uncertainty analysis under UUT Repeatability. After the data points are taken, the curve fit of the ITS-90 sub range 7 of the calibration is checked by evaluating the residuals using a Chi-Squared analysis (since an extra point is measured for an over-determined curve fit).

In addition to the quality check limits applied to the UUT, a check standard PRT is included in each calibration run. Control charts of the check statistics are maintained to ensure the calibration process is in control. The check standard variability is established in the uncertainty analysis under Process Variability. Process Variability is the allowed standard deviation limit for the check standard statistics at each temperature.

5. UNCERTAINTY ANALYSIS

The cal lab had extensive experience calibrating PRTs in stirred-liquid baths but calibration by comparison in dry-blocks was a new method that was approached cautiously. Uncertainties such as Measurement Precision, SPRT

Calibration, SPRT Drift, Readout Accuracy and Insulation Leakage were well understood since they were based on work done in the stirred-liquid bath calibration processes.

1.1 Uncertainty Due to Axial and Radial Gradients

The two sources of uncertainty in dry-block measurement which concerned the lab most were the axial (vertical) and radial (hole-to-hole) gradients (temperature uniformity). Since axial and radial gradients are typically sources of relatively large temperature errors in dry-blocks, the cal lab decided to use the Fluke, Hart Scientific model 9173 high-temperature dry-block calibrator with axial gradient control for the temperature range 157 to 660 °C (the temperatures at which good uniformity is the most difficult to achieve). The Fluke, Hart Scientific model 9170 peltier-cooled, dry-block calibrator was chosen to for the range -40 to 0 °C.

The cal lab team performed several measurements to understand the gradient effects in the dry-blocks. The radial gradients were found to be ±30 mK or less (between the center hole and each UUT hole) over the range 157 to 660 °C in the hot dry-block (9173) and ±10 mK or less in the cold dry-block (see Figure 3). It is important to note that since the axial gradients affect the radial gradient measurements, axial gradient adjustment is performed before measuring radial gradients. Also, due to the design of the 9173 dry-block, the team found that radial gradients can be optimized by turning the insert inside the dry-block to find a position of lowest radial gradient for all measurement wells.

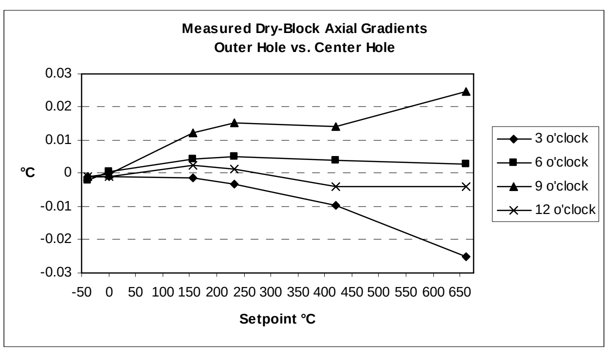

Fig. 3. Measured Gradient Data

The axial gradients were found to stay within ±50 mK in the bottom 40 mm of both the hot and cold dry-block units over the range -40 to 660 °C if gradient adjustment is performed before each calibration run on the hot unit (cold unit has no gradient adjustment). The team is continuing to study the repeatability of the axial gradient to determine if the axial gradient adjustment can be done over longer intervals rather than before each run.

Using the entire ±50 mK axial gradient tolerance in the uncertainty analysis would not be adequate since the overall calibration uncertainty is required to be ±50 mK or less. The axial gradient spec alone would consume the entire uncertainty budget. To reduce the effect of the axial gradient, the midpoint of the sensors in the UUTs and SPRT are vertically aligned. However, since sensors are not always placed inside the sheath at the same distance from the tip and differences in construction between the SPRT and UUT, it is difficult to know the exact midpoint.

Manufacturer specifications and experimental measurements indicated that the UUT sensors could be aligned within ±5 mm of the SPRT sensor using 5 mm alumina spacers in the bottom of the UUT measurement wells. Since the axial gradient is fairly linear over the bottom 40 mm, the uncertainty due to the axial gradient was estimated to be ±6.25 mK. This number is derived by first dividing the ±50 mK gradient allowance by the size of the gradient region (40 mm) to result in a gradient characterization of 1.25 mK/mm. Since the sensors could be aligned within ±5 mm, multiplying ±5 mm by 1.25 mK/mm results in a gradient error of ±6.25 mK.

1.2 Other Uncertainty Information

The complete calibration uncertainty analysis is listed in Table 3. It is important to note that the uncertainties are based on an SPRT (Fluke, Hart Scientific model 5699) used as the reference thermometer. Since SPRTs are too fragile for some field calibration environments, it may be necessary to use a more rugged PRT as the temperature reference. For this purpose, a second uncertainty analysis based on the Fluke, Hart Scientific model 5628 PRT is provided in Table 4. The resulting changes in the uncertainty analysis are indicated in the shaded region.

It should be noted as well that the 5628 allowed drift at 0 °C is ±10 mK. This is controlled by monitoring the 5628 at the triple-point of water. Otherwise, using the manufacturer's estimated long-term drift would add significantly more uncertainty. Experience has proven that if the 5628 is well taken care of, the ±10 mK drift allowance is very reasonable. Also, if the system is to be used in a field calibration application the additional effects due to environmental conditions may need to be accounted for.

Uncertainty Sources:	Type	-197 °C mK	-38 °C mK	0 °C mK	157 °C mK	232 °C mK	420 °C mK	660 °C mK
Process Variability	Norm	3.0	3.0	3.0	5.0	5.0	5.0	6.0
UUT Precision	Norm	2.8	2.8	2.8	2.8	3.3	4.4	5.6
Ref. SPRT Precision	Norm	2.8	2.8	2.8	2.8	2.8	2.8	5.6
Ref. SPRT Calibration	Norm	0.5	0.2	0.1	0.6	0.5	0.6	1.1
Ref. SPRT Drift	Rect	0.4	1.7	2.0	3.2	3.8	5.1	6.7
Radial Uniformity	Rect	2.0	10.0	10.0	30.0	30.0	30.0	30.0
Axial Uniformity	Rect	2.0	6.3	6.3	6.3	6.3	6.3	6.3
Readout (SPRT)	Rect	0.1	0.2	0.3	0.4	0.5	0.7	0.9
Readout (UUT)	Rect	0.4	1.3	1.5	2.5	3.1	4.4	6.3
Insulation Leakage	Rect	10.0	10. 0	10.0	10.0	10.0	10.0	10.0
UUT Repeatability	Norm	0.6	2.8	3.3	5.3	6.3	8.6	11.2
Total (k=2):		**14.6**	**20.6**	**21.0**	**40.7**	**41.5**	**43.9**	**47.9**

Table 3. Uncertainty analysis with SPRT as reference (Fluke, Hart Scientific Model 5699)

Uncertainty Sources:	Type	-197 °C mK	-38 °C mK	0 °C mK	157 °C mK	232 °C mK	420 °C mK	660 °C mK
Process Variability	Norm	3.0	3.0	3.0	5.0	5.0	5.0	6.0
UUT Precision	Norm	2.8	2.8	2.8	2.8	3.3	4.4	5.6
Ref. PRT Precision	Norm	2.8	2.8	2.8	2.8	2.8	2.8	5.6
Ref. PRT Calibration	Norm	3.0	5.0	2.5	3.0	3.0	4.5	7.0
Ref. PRT Drift	Rect	1.8	8.4	10.0	16.0	18.9	25.7	33.7
Radial Uniformity	Rect	2.0	10.0	10.0	30.0	30.0	30.0	30.0
Axial Uniformity	Rect	2.0	6.3	6.3	6.3	6.3	6.3	6.3
Readout (Ref. PRT)	Rect	0.1	0.2	0.3	0.4	0.5	0.7	0.9
Readout (UUT)	Rect	0.4	1.3	1.5	2.5	3.1	4.4	6.3
Insulation Leakage	Rect	10.0	10. 0	10.0	10.0	10.0	10.0	10.0
UUT Repeatability	Norm	0.6	2.8	3.3	5.3	6.3	8.6	11.2
Total (k=2):		**15.9**	**24.8**	**24.3**	**44.9**	**47.1**	**53.4**	**62.7**

Table 4. Uncertainty analysis with PRT as reference (Hart Scientific Model 5628)

6. CALIBRATION PROCESS VERIFICATION

In order to introduce the new dry-block calibration process into the cal lab, the team performed an inter-comparison between the new process and an established process with E_n results of less than 0.5 at each temperature point. E_n is a statistical analysis that combines measurement error versus measurement uncertainty of both calibration processes as a ratio. A resulting ratio of 1 or less indicates that measurement error is consistent with the combined uncertainties. The equation is:

$$E_n = \frac{Error}{\sqrt{U_1^2 + U_2^2}}$$

Where *Error* is the measured difference between the two calibration processes and U_1 and U_2 are the uncertainties from each process.

7. FUTURE IMPROVEMENTS

The cal lab has continued to monitor the new process by cross-checking calibrations with the previously established stirred-liquid bath processes. So far, the results have been very good but the team has noticed some trends in the data that could help improve the calibrations. With further study it may be possible to reduce the source of uncertainty associated with radial gradients. Preliminary results indicate that the gradients are fairly repeatable and could possibly be reduced with a correction applied to each well. Another future improvement the cal lab would like to investigate is the possibility of increasing calibration capacity by adding additional measurement holes in the dry-block inserts.

8. CONCLUSION

Fluke’s Hart Scientific cal lab was able to create a new calibration process base on dry-block heat sources that resulted in a maximum calibration uncertainty of 50 mK or less over the range -197 to 660 °C. It was demonstrated that with careful measurements and a comprehensive uncertainty analysis, it is possible to achieve reliable, low-uncertainty measurements, even in the field.

9. ACKNOWLEDGEMENTS

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