Improvement Study on the Use of Dry-Well Calibrators for PRT Calibration

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Abstract

Radial and axial temperature gradients in the block and instability and hysteresis of the control sensor are main deficiencies that affect the performance of dry-well (block) calibrators. These inherent limitations are inevitable in a dry-well calibrator with single-zone control. In order to reduce calibration errors and improve the performance of dry-well calibrators, a new dry-well calibrator with dual-zone control, called "Metrology Well Calibrator," was developed at Fluke-Hart Scientific. The performance of the new dual-zone dry-well calibrator was evaluated.

The axial temperature uniformity was measured using a specially made platinum resistance thermometer. The special PRT has a very short sensor within a platinum capsule, but no sheath. It is an excellent probe for measuring vertical temperature uniformity. The design of this PRT will be described briefly.

Several PRTs with different structures were calibrated at fixed points and then tested in the dual-zone Metrology Well Calibrator to identify measurement errors. The results will be reported.

1. Introduction

Dry-well calibrators are widely used as temperature standards or comparison apparatus in many secondary level calibration laboratories and in various industrial fields. Since dry-well calibrators are designed primarily for convenience and efficiency in a calibration process, and since they are usually single-zone controlled, some inherent limitations are inevitable. In order to reduce calibration errors and improve the performance of dry-well calibrators, a new dry-well calibrator with dual-zone control, called "Metrology Well Calibrator", was developed at Fluke-Hart Scientific.

Several common operational mistakes by users of dry-well calibrators were noticed in laboratories. As a result of incorrect use, large calibration errors were reported by many users. Usually, platinum resistance thermometers (PRTs), thermocouples, thermistors, and other industrial temperature probes can be calibrated in a dry-well calibrator. However, the calibration uncertainties when calibrating different units under test (UUTs) using the same reference thermometer and the same readout instruments may vary if an incorrect method is used. The experimental results indicated that to achieve the lowest calibration uncertainty, the method should be adjusted somewhat depending on the design of the UUTs.

It is well known that the axial temperature uniformity of a dry-well calibrator is generally worse than that of a liquid bath. Axial temperature nonuniformity can be one of the main contributors to calibration error. Traditionally, the temperature uniformity is measured by a PRT, an SPRT, or a thermocouple. However, due to the long element length of a PRT or an SPRT, or stem conduction heat loss or inhomogeneity of thermocouples, the axial temperature uniformity usually cannot be measured accurately. To better measure the axial temperature uniformity, a special platinum resistance thermometer was constructed. The special PRT has a very short sensor (about 5 mm) within a platinum capsule, but with no sheath. It is an excellent probe for measuring axial temperature uniformity. The design of this PRT will be described later in the paper.

Using the special PRT, along with eight PRTs with different element lengths and designs, a series of studies was made. These experiments included evaluating the axial temperature uniformity, radial temperature uniformity, and the effects of the hysteresis of the control sensor in several different dry-well calibrators.

Because poor axial temperature uniformity in a dry-well calibrator can cause large errors when it is used for calibration, one method to reduce the effect of axial temperature nonuniformity is to build a dry-well calibrator with an adjustable axial temperature gradient. A dual-zone dry-well calibrator, the Metrology Well Calibrator, manufactured by Fluke-Hart Scientific, is introduced in this paper, and the test results of its axial temperature uniformity are presented. Several PRTs with different structures were calibrated at fixed points and then tested in the Metrology Well Calibrator to identify measurement errors. The results will be reported.

A few dry-well calibrators with different designs and temperature ranges made by different manufacturers were tested. This paper presents test results for one of these dry-well calibrators, to demonstrate test methods, typical errors, and proposed proper techniques of use. Test results might be different among different dry-well calibrators, but the test methods described in this paper can be applied to all types.

Besides the inherent deficiencies of dry-well calibrators, correct use is also critical to minimize calibration errors. Some common operational mistakes and measurement errors were discussed in a previous paper published at MSC by Mike Hirst [7].

2. Experimental Apparatus

A few apparatus were selected for the experimental study. Hart Scientific Model 1590 "Super-Thermometer," in conjunction with a Model 2590 multiplexer, was used as the electrical measurement instrument. Its measurement uncertainty for resistance ratios is 1 ppm. An external 100-ohm standard resistor was used as the reference resistance. The standard resistor was maintained in an oil bath at 25°C.

A few dry-well calibrators made by different manufacturers, including the Metrology Well Calibrator made by Fluke-Hart Scientific, were tested in this study.

Ten platinum resistance thermometers were used in the experiments. Their structures and element lengths varied. Eight of the PRTs are available commercially. Two identical PRTs were specially made for the experiments. They have very short sensors within platinum capsules, but no sheaths. The element length is only 5 mm. The short sensor makes it an ideal probe for measuring axial temperature uniformity. The structure of the PRT is shown in Figure 1. It has 10 ohms of resistance near 0°C and a temperature range from 0°C to 1100°C. The thermometer adopts SPRT design techniques, being strain free and using high-purity platinum wire. A platinum protection capsule shields and holds the resistance element. The element was sealed permanently using high-temperature sealant. The thermometer has four lead wires to eliminate lead resistance errors. The four wires are insulated by a thin-walled single-bore alumina tube separated by alumina disks. The thermometer was calibrated in fixed points over the range from 0°C to 961.78°C.

All PRTs involved in the study were calibrated in fixed-point cells, including the triple point of water. After experiments were completed, their resistances at the triple point of water (Rtp) were measured again. All PRTs were shown to be sufficiently stable throughout the experiments.

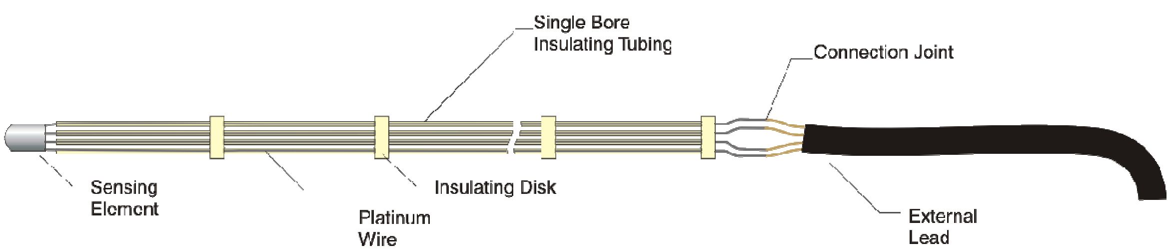

Figure 1, Structure of the platinum resistance thermometer for temperature uniformity measurement

3. Test Results and Discussion

3.1 Axial Temperature Uniformity

The axial temperature uniformity of a dry-well block calibrator with single-zone control was measured using four PRTs with different element lengths. The method of measuring the axial uniformity was described in our previous paper [1]. The total depth of the thermometer wells of the single-zone dry-well calibrator was 150 mm, while that of the dual-zone Metrology Well calibrator was 203 mm. The axial temperature uniformity was measured in the range from the bottom of the well (0 mm) up to 60 mm for the single-zone dry-well calibrator and up to 80 mm for the Metrology Well Calibrator. Typically, PRT elements are shorter than 50 mm. The tests showed that the measurement results are different when using different PRTs that have different element lengths, even though the measurements were made in the same dry-well calibrator and at the same temperature (see Figure 2).

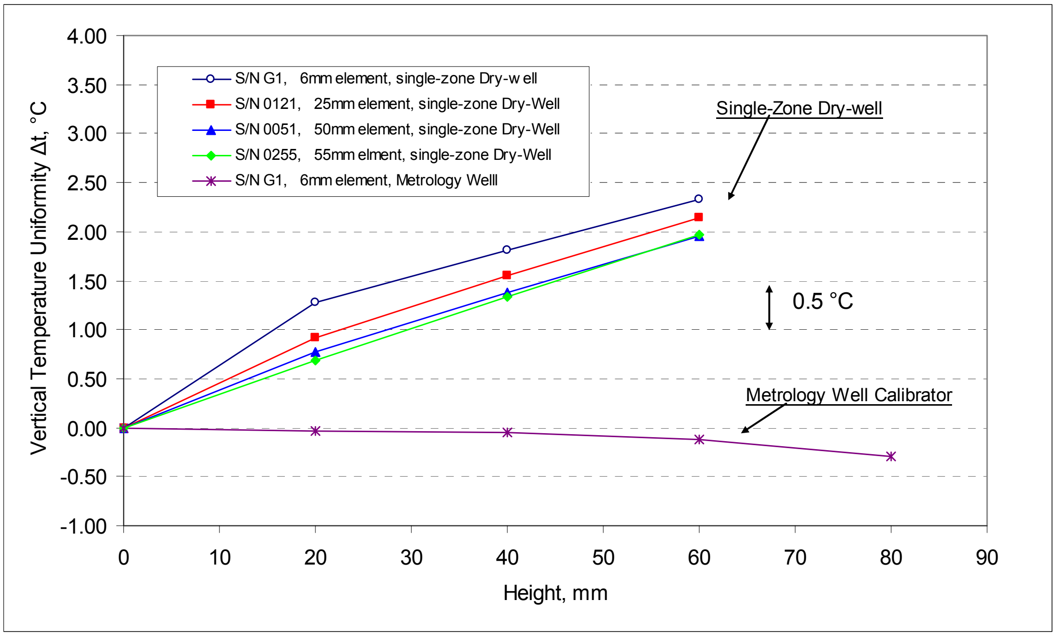

Figure 2, Axial temperature uniformity using different PRTs with different element lengths at 660°C.

With the single-zone dry-well calibrator, as the PRT was moved up from the bottom, the measured temperature increased. It was noticed that the temperature uniformity appeared better when using a PRT with a longer element than when using a PRT with a shorter element. The PRT with serial number G1 is the specially made short element thermometer with no sheath. The axial temperature uniformity measured by this PRT was the worst. The true temperature uniformity is measured more accurately with this probe than by the other three PRTs. The longer elements tend to average the temperature over their lengths.

The axial temperature uniformity of the Metrology Well Calibrator was also measured. Comparing the results in Figure 2, it is clear the axial temperature uniformity of the dual-zone Metrology Well Calibrator is much better than that of the single-zone dry-well calibrator. Axial temperature uniformities of the Metrology Well Calibrator at other temperatures are shown Figure 3.

3.2 Radial Temperature Uniformity

Radial temperature uniformities were measured using the two specially made PRTs, S/N G1 and S/N G2, described above. The method of measuring the radial temperature uniformity was described in our previous paper [1]. The test results are shown in Table 1. The results show that the radial temperature uniformities of both dry-well calibrators are much better than the axial temperature uniformities. Error introduced by radial temperature uniformity is only a small part of the total calibration error. The radial temperature uniformity of the Metrology Well Calibrator was found to be slightly better than that of single-zone dry-well calibrator.

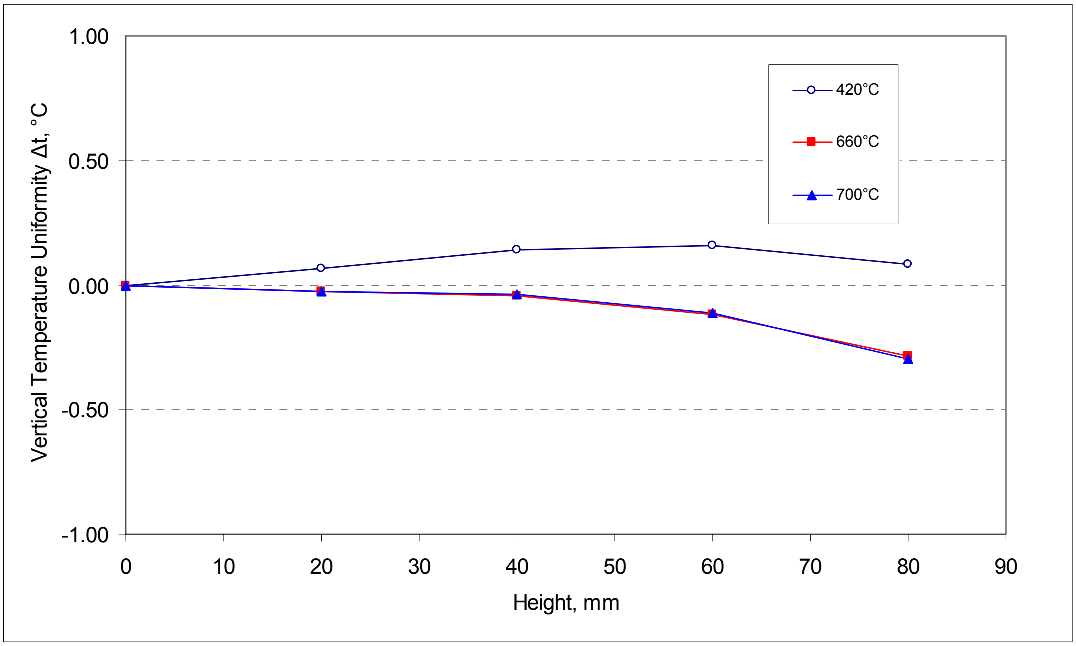

Figure 3, Axial temperature uniformity of Metrology Well Calibrator at different temperature.

Table 1, Radial temperature uniformity

	Well No.	#1	#2	#3	#4
Single-zone Dry-well	Δt at 661°C	+0.000°C	+0.035°C	-0.040°C	0.018°C
Dual-zone Metrology Well	Δt at 661°C	+0.000°C	-0.026°C	-0.030°C	-0.001°C
Dual-zone Metrology Well	Δt at 420°C	+0.000°C	-0.017°C	-0.022°C	-0.021°C
Dual-zone Metrology Well	Δt at 232°C	+0.000°C	+0.008°C	-0.006°C	-0.004°C

3.3 Hysteresis of the Control Sensor

When the dry-well's display temperature is used as the reference to calibrate thermometers, hysteresis of the internal sensor may affect the calibration uncertainty. Hysteresis in a dry-well calibrator is observed when the actual temperature at which the dry-well calibrator stabilizes at a given set-point is different depending on whether the temperature was reached by heating the block from a low temperature or cooling it from a high temperature. The nature and evaluation methods of hysteresis in platinum resistance thermometers have been presented by D. J. Curtis[2]. A test method to measure the hysteresis effect was described in our pervious paper [3]. Since the control sensor for a dry-well calibrator is usually a lower-cost industrial platinum resistance thermometer (IPRT), it is likely to exhibit hysteresis.

To measure the effect of hysteresis on the performance of the dry-well calibrators, two SPRTs and one PRT, each with no detectable inherent hysteresis, were used in the experiment. The thermometers were first measured at the triple point of water. Next they were inserted into the dry-well calibrator at room temperature. The temperature of the dry-well calibrator was raised to 330°C, near the midpoint of the temperature range. After stabilizing for two hours, temperature measurements from the three probes were taken. Then the temperature of the dry-well calibrator with the three thermometers was raised to 660°C. After stabilizing for two hours again, temperatures were measured. This is the "increasing half-cycle." Then the dry-well calibrator was cooled down to 330°C. After it was allowed to stabilize for two hours, the readings were taken again. This is the "decreasing half-cycle." After this measurement, the thermometers were taken out of the dry-well calibrator and measured at the triple point of water again to make sure they did not drift significantly. The temperatures of the thermometers at 330°C for the two half-cycles were compared.

The test results for the single-zone dry-well calibrator are shown in Figure6. The hysteresis measured by the three thermometers was 0.046°C, 0.047°C, and 0.050°C respectively.

The producers of the Metrology Well Calibrator desired to optimize the performance of their instrument, particularly with regard to hysteresis, by developing a new sensor that was rugged, able to handle temperatures to 700°C, and had low hysteresis. Test results for the Metrology Well Calibrator using this improved sensor are also shown in Figure 6. Hysteresis was found to be about 0.02°C. Data collected during repeated cycling, showing additional information regarding hysteresis in this instrument, are shown in Figure 7.

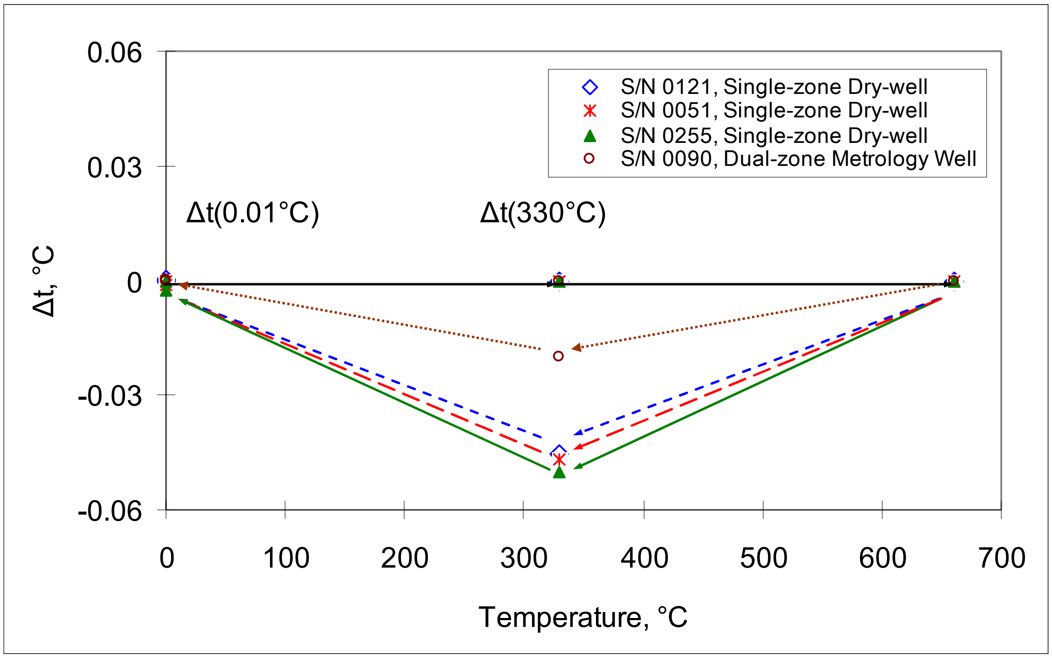

Figure 6, Effect of Hysteresis of the Control Sensor

If the display of the dry-well calibrator is used as the reference temperature (internal reference) during calibration of a UUT sensor, hysteresis of the dry-well calibrator's sensor might cause significant errors. When an external reference thermometer is used to calibrate the thermometers, the hysteresis of the dry-well calibrator's internal sensor will not contribute to the calibration uncertainty. However, any hysteresis in the reference thermometer or the UUT sensor will cause errors. To minimize the effects of hysteresis in the dry-well calibrator, reference thermometer, or UUT, consider measuring the UUT twice at each calibration temperature, once during the increasing half-cycle and again during the decreasing half-cycle, and using the average of the two measurements. However, if the application for the UUT is understood to operate the device consistently in one direction only, say always after heating from room temperature, then perhaps it might be better if the calibration used measurements of just that half cycle.

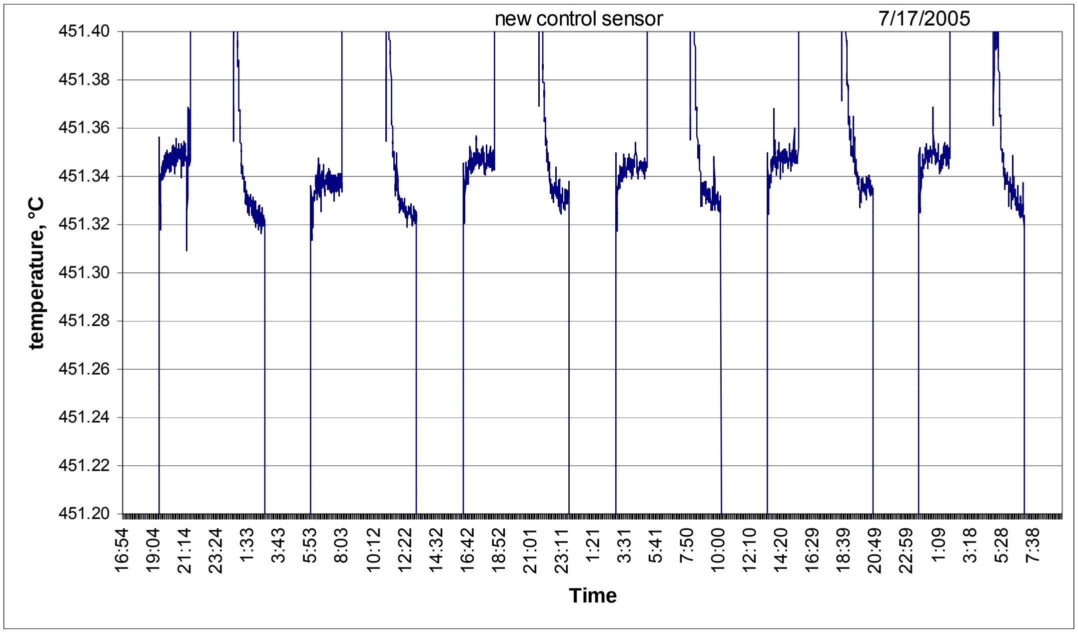

Figure 7, Stability of the control sensor during thermal cycles

3.4 Other Possible Calibration Errors

For a single-zone dry-well calibrator, it is necessary for the thermometer diameter to be matched to the thermometer well size. It is suggested that the gap between the thermometer and the well should be around 0.01 inch (0.254 mm) [5]. If this gap is too large, the calibration error could be significant because of stem conduction. A simple experiment of size matching was made, with results shown Figure 8. A PRT with 0.25 inch (6.35 mm) diameter was calibrated in a dry-well calibrator using an external reference thermometer at 660°C. When the thermometer was inserted into the proper thermometer well, i.e. the gap between the thermometer sheath and the well was 0.01 in. (0.254 mm), the calibration error was +0.03°C. When the thermometer was inserted into a 0.3125 in. (7.938 mm) well and a 0.375 in. (9.525mm) well, the calibration errors were larger, at -0.05°C and -0.22°C respectively. This shows the importance of keeping the thermometer sheath diameter and the well size matched.

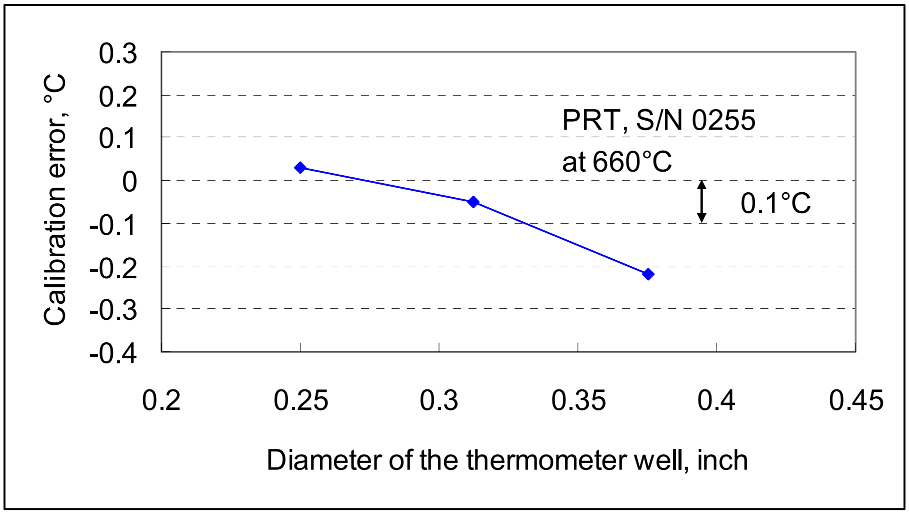

Figure 8, Measurement errors when the diameter of the PRT does not match the well diameter

An investigation done at Fluke-Hart Scientific indicated the calibration error in the dry-well calibrator was significantly related to thermometer immersion (stem effect) [6] [7]. Experiments on immersion depth were conducted in a specially fabricated dry-well block that was immersed into a salt bath at 420°C to minimize the actual gradient in the block. No bath fluid was able to enter the well. A wide range of thermometers was tested. The thermometers ranged from high quality industrial PRTs to laboratory quality SPRTs. The thermometer wells were 8.5 inches (216 mm) deep, and diameters were provided to match each of the thermometer diameters. All of the thermometers were normalized at the maximum immersion depth. Each thermometer was then withdrawn 1 cm at a time and data taken at each depth. The data was then plotted on a graph representing the deviation in temperature versus depth of immersion. Since each thermometer had its own variation in diameter, sensor length, and stem conduction properties, each curve was slightly different. Temperature deviations were then compared at different common well depths for a variety of dry-well calibrators. Immersion depths of 6 inches (152 mm), 4.9 inches (125 mm), and 4 inches (102 mm) were compared. The average spread of temperatures at 6 inches was 0.03°C, at 4.9 inches it was 0.08°C, and at 4 inches it was 0.20°C. The range of values at each depth was measured against a PRT with a 5 mm long sensor and known to have low stem conduction.

For the Metrology Well Calibrator, due to the excellent axial temperature uniformity and greater-than-typical immersion depth of 8 inches (203 mm), the effect of immersion depth on the calibration errors is typically small. This can be confirmed by comparison of calibration results using probes with different element lengths, which will be discussed following.

3.5 Calibration Using the Single-Zone Dry-Well Calibrator

To see what typical calibration errors might be when using a single-zone dry-well calibrator, including effects of axial and radial temperature gradients and stem conduction, calibrations of several PRTs were performed using the single-zone dry-well calibrator and a reference thermometer. First, four PRTs with the same element lengths were tested. The PRTs were previously calibrated in fixed-point cells to ensure they could accurately measure the same temperature (in a deep, uniform well). Then the PRTs were measured in the single-zone dry-well calibrator at a temperature near 660°C. The measured temperatures of the PRTs in the dry-well calibrator are shown in Figure 9. The maximum difference was slightly less than 0.1°C. One would expect that, since the PRTs were of similar construction, differences due to axial temperature nonuniformity and stem conduction would be small so most of the error is due to radial temperature nonuniformity.

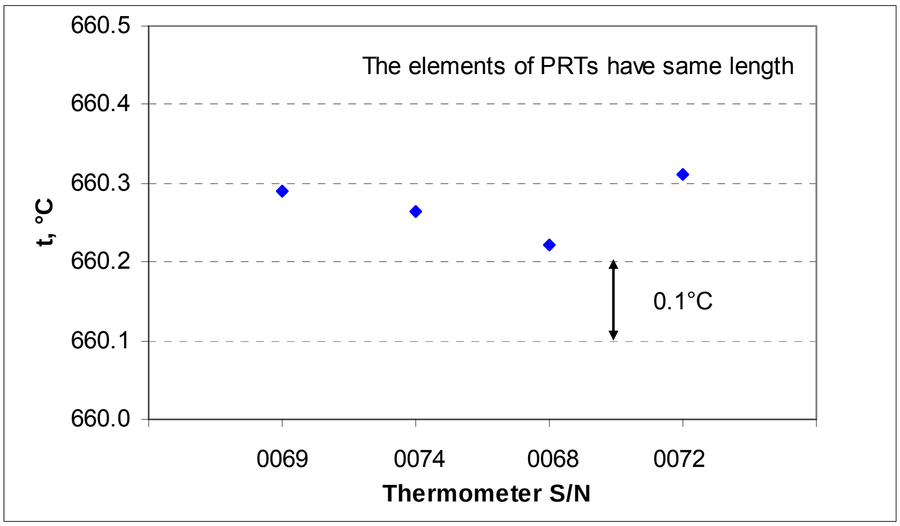

Figure 9, Comparison calibration of PRTs with same element length at 660°C in a single-zone dry-well calibrator

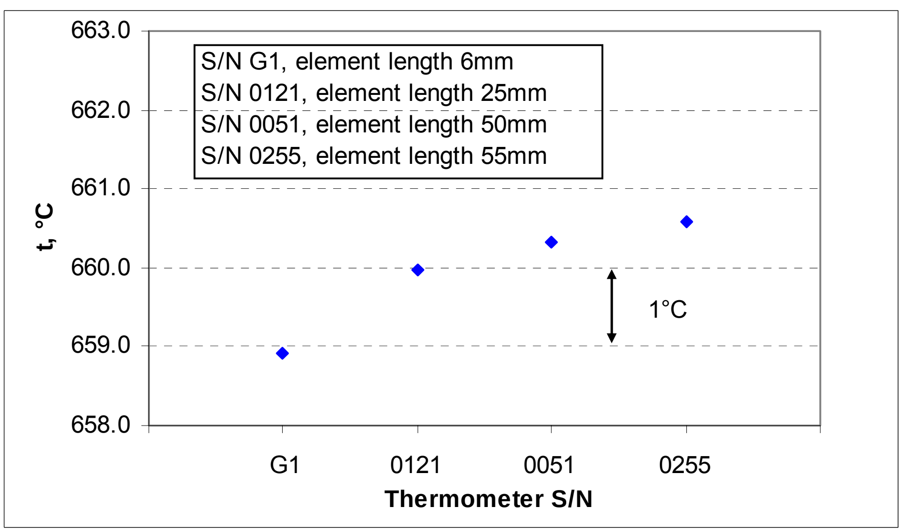

Figure 10, Comparison calibration results of PRTs with different element lengths at 660°C

When the PRTs had different element lengths, the calibration errors were much larger. The results of comparing PRTs having different element lengths at 660°C are shown in Figure 10. These also were previously calibrated in fixed-point cells for consistent accuracy. The differences between measurements in the dry-well calibrator were as large as nearly 2°C. The tight fit of the probes in the wells and their immersion depth precluded stem conduction from causing such large errors. It can only be concluded that the large errors are primarily due to the effect of axial temperature gradient. To attempt to reduce these errors, the PRTs with shorter elements were moved slightly higher up in the wells so that the all the midpoints of the elements were at the same axial position. The errors using this technique were significantly less, as shown in Figure 11. The maximum difference was reduced from about 2°C to less than 0.2°C. Of course, use of this method requires understanding of the construction of the PRTs so that the locations of the midpoints of the sensor elements can be identified.

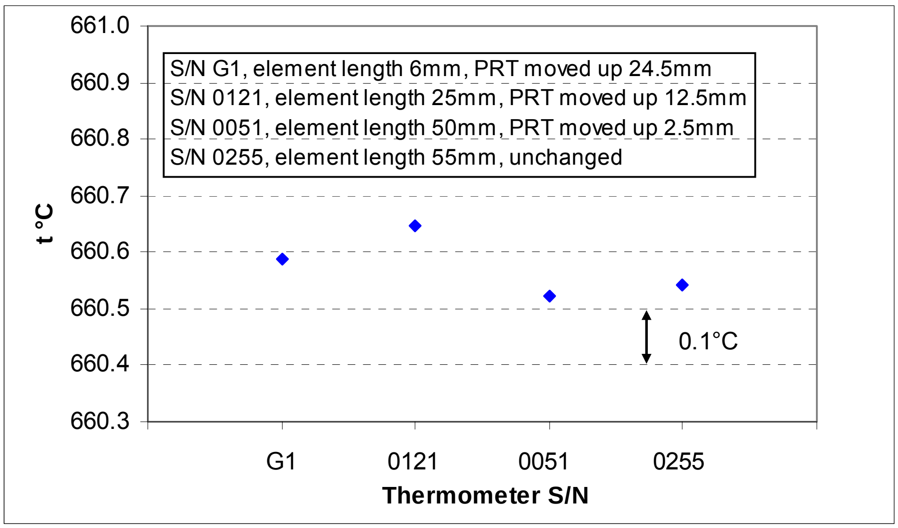

Figure11, Comparison calibration of PRTs with different element lengths but with mid-points at the same level

3.6 Calibration using the Metrology Well Calibrator

Similar PRT comparison tests were performed with the Metrology Well Calibrator. Eight precision PRTs and SPRTs with different element lengths were used in the experiment. All PRTs were calibrated by fixed-point cells in the range from 0.01°C to 660.323°C. The calibration uncertainties at 231.928°C, 419.527°C, and 660.323°C are 0.006°C, 0.009°C, and 0.014°C respectively. The sensor element lengths of the probes are all less than 55mm, but vary. Results of comparing each of seven PRTs against the eighth at three temperatures are shown in Table 2.

Table 2, PRT comparisons in the Metrology Well Calibrator (differences in °C)

Temperature Set Points	UUT #1	UUT #2	UUT #3	UUT #4	UUT #5	UUT #6	UUT #7
Element length	50 mm	6 mm	50 mm	25 mm	50 mm	55 mm	45 mm
660°C	+0.029	-0.008	+0.017	-0.010	+0.030	-0.076	-0.008
420°C	+0.015	-0.007	+0.011	-0.004	+0.020	+0.024	-0.024
232°C	+0.008	-0.005	+0.012	-0.006	+0.012	+0.015	+0.007

The total expanded uncertainties (k=2) for the Metrology Well Calibrator when using it as a temperature reference—which include errors associated with control sensor accuracy, hysteresis, and drift; control instability; axial and radial nonuniformity; and thermal loading—is estimated to be 0.25°C at 660°C, 0.2°C at 420°C, and 0.15°C at 250°C respectively [8]. Uncertainties when using an external reference thermometer are much lower since the control sensor related errors are excluded, and other errors, such as due to axial nonuniformity and thermal loading, are reduced.

4. Conclusions

For a single-zone dry-well calibrator, the calibration uncertainty can be improved significantly using correct methods and procedures. Usually, the axial temperature is the most significant of all the calibration error sources. However, by keeping the midpoints of the elements of all thermometers at the same level, it is possible to significantly reduce calibration errors introduced by the axial temperature nonuniformity. It is very important to correctly use the dry-well calibrator, since incorrect operation can introduce large errors into the calibration. For the lowest calibration uncertainty, it is suggested a reference thermometer should be used during calibration.

The dual-zone Metrology Well Calibrator has much better performance compared to typical single-zone dry-well calibrators. The biggest improvement is the excellent axial temperature uniformity, which is usually the most significant contributor to the calibration error in single-zone dry-well calibrators.

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