STUDY OF A NEW FIXED-POINT SYSTEM FOR CALIBRATION OF SHORT SECONDARY PLATINUM RESISTANCE THERMOMETERS

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Abstract - A new fixed-point system was developed to calibrate short (8 – 9 inches) secondary platinum resistance thermometers (PRTs) and industrial platinum resistance thermometers (IPRTs). The system includes a re-designed metal-cased fixed-point cell based on an existing metal-cased fixed-point cell which was recently developed to calibrate secondary PRTs (12 inches) at Fluke – Hart Scientific, and a maintenance furnace with a three-zone controller that provides excellent vertical temperature uniformity. The design and structure of the new fixed-point system is described in the paper. The system was tested with tin and zinc freezing-point cells by realizing the freezing plateau. The comparison testing was carried out with the standard size fused quartz glass shell tin and zinc cells. The testing results are presented, and the freezing plateau is fully evaluated and discussed in the paper.

1. Introduction

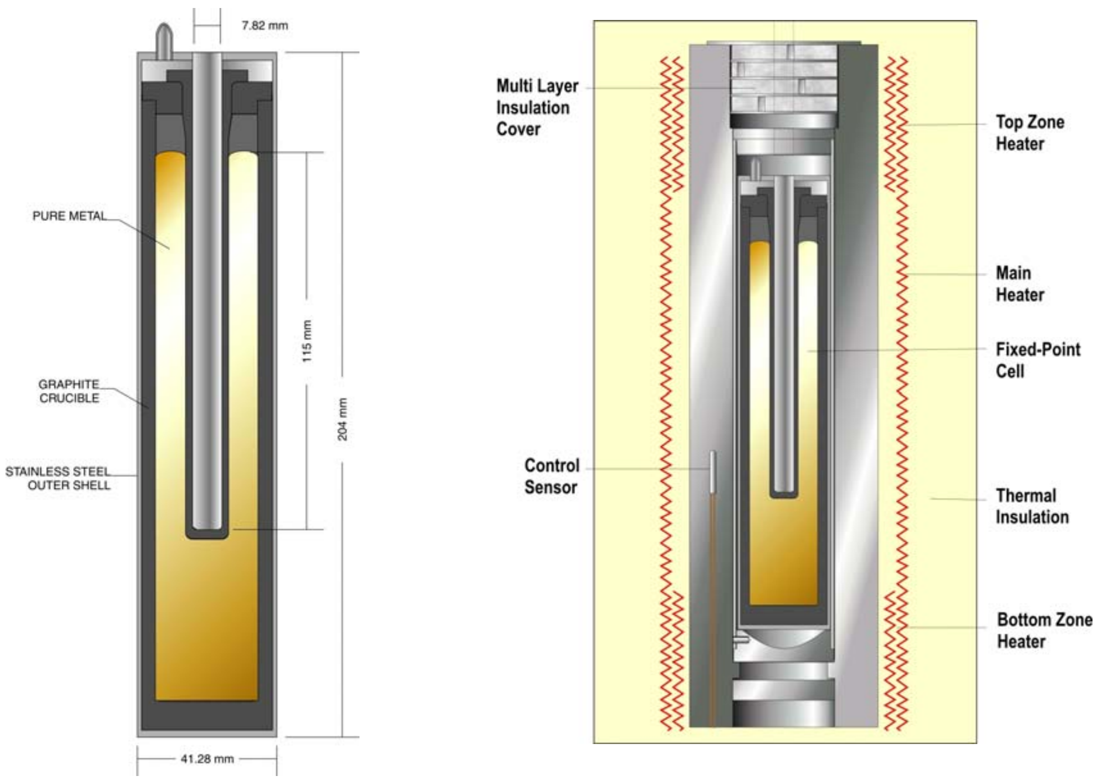
Short (8 - 9 inches) secondary platinum resistance thermometers (PRT) are widely used in many fields, but these short PRTs cannot be calibrated in the normal size fixed-point cell systems because of their short stems. A specially designed fixed-point system is needed to calibrate these short PRTs to the lowest levels of uncertainty. This system should include specially designed fixed-point cells and a maintenance furnace which can hold the short stem PRTs. Because of the short stems of these PRTs, the PRT sensors are close to the top of the maintenance furnace. This results in larger heat loss from the probe stem and the sensor. Ensuring a suitable immersion depth of the short PRT is very important in order to minimize the influence of the heat loss through the probe on the measurement result. The immersion depth of the probe sensor should be as large as possible in order to reduce the heat loss of the sensor during testing.

The furnace's vertical temperature uniformity also has a very important influence on the short PRT's calibration because of the relatively shorter immersion depth and the larger heat loss at the top of the furnace. In order to obtain a good uniform temperature in the furnace work zone, a three-zone controller should be used to maintain the furnace temperature. Because of the larger heat loss of short PRTs at the top of the furnace, a special furnace top cover should be used to minimize the heat loss, especially radiation heat loss from the top of the furnace.

A fixed-point system for calibration of short PRTs was recently designed and developed at Fluke Corp. – Hart Scientific Division. The system includes two maintenance furnaces, a specially designed metal-cased tin fixed-point cell and a metal-cased zinc fixed-point cell. The system was tested by realizing the freezing plateaus of the cells and comparing their temperatures with those of standard size fused quartz glass shell fixed-point cells. The freezing plateau results and the comparison testing results are presented in this paper, and the uncertainties of the new system are analyzed.

2. Fixed-point system

A schematic drawing of the fixed-point system designed for calibration of short PRTs is shown in Figure 1. The system contains a maintenance furnace and a metal-cased fixed-point cell. It can be difficult to obtain a uniform temperature distribution in the fixed-point work zone inside such a small furnace. In order to adjust the furnace temperature gradient conveniently, three independently controlled heaters are used in the furnace. One main heater is used to control the whole furnace work zone, a second heater controls the bottom zone, and a third controls the top zone. The top zone and the bottom zone can be adjusted independently to maintain a uniform temperature profile. In order to minimize the heat loss due to radiation at the top of the furnace, a multilayer insulation cover was designed and used as shown in Figure 1 (b). The structure of the fixed-point cell was designed based on the existing metal-cased mini fixed-point cells (Fluke Corp. – Hart Scientific Division 594x series mini metal-cased fixed-point cells) with a change to the cell's overall length and reentrant well length [1]. The existing mini fixed-point cells developed at Fluke Corp. – Hart Scientific Division have been thoroughly tested and compared with the standard size fused quartz glass shell fixed-point cells [2]. The reentrant well length of the short cells is 145 mm, and the immersion depth is 115 mm. Both the reentrant well length and the immersion depth of the short fixed-point cells are noticeably shorter than that of the existing mini fixed-point cells. The influence of the reduction of the immersion depth on the duration of the freezing plateau will be analyzed and discussed in this paper.



(a) (b)

Fig. 1. Schematic drawing of the fixed-point cell (a) and the fixed-point system with the fixed-point cell and the maintenance furnace (b).

3. Test methods

The system was tested using the short metal-cased tin and zinc fixed-point cells to realize the freezing plateaus. In order to evaluate the performance of the short fixed-point cells, comparison testing was carried out against two standard size tin and zinc fixed-point cells.

3.1 Furnace temperature gradient

Before testing, the furnace vertical temperature gradient was carefully adjusted. Because of the short immersion depth, a specially designed short sensor platinum resistance probe was made to test the furnace vertical temperature gradient. The adjusted vertical temperature profiles of the two furnaces are shown in Figure 2. Temperatures at the bottom and top tend to be lower because of greater heat loss out the ends. The top is particularly susceptible to heat loss because of the greater exposure. In order to reduce the heat loss from the top, a special cover with a sandwich structure consisting of alternating layers of stainless steel and ceramic fiber insulation was designed and used in the testing. It can be seen that the largest temperature difference is ± 0.065 °C for the first furnace (for tin cell testing), and ± 0.070 °C for the second furnace (for zinc cell testing). Subsequent observation of the freezing plateaus showed that the temperature gradients were adequately low for testing the short tin and zinc fixed-point cells.

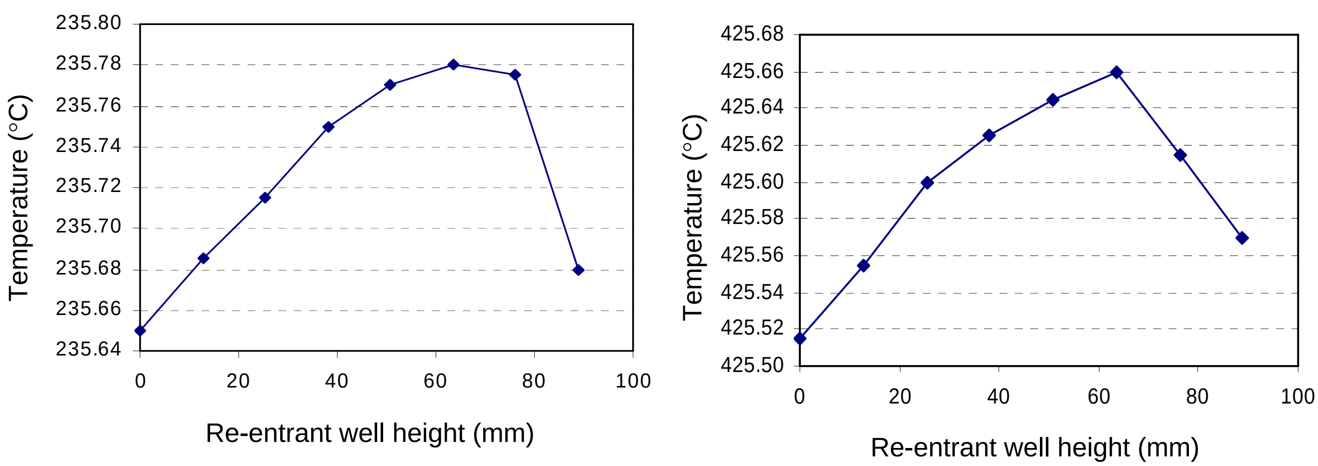


Fig. 2. Vertical temperature profiles of the tin and zinc cell furnaces.

3.2 Realizing the freezing plateau

Two short metal-cased tin and zinc fixed-point cells were used to realize freezing plateaus in order to test the performance of the system. To prepare for freezing the tin cell, we first raised the temperature of the furnace, with the cell in place, to 235 °C and let it stabilize overnight. Then to initiate the test the next day, the furnace's temperature was dropped to 229 °C at a slow scan rate of 0.1 °C/minute. When recalescence was observed, two silica quartz glass tubes were inserted into the cell's reentrant well for four minutes each to induce nucleation. After induction, the furnace temperature was set to 231.5 °C, which is 0.4 °C below the theoretical freezing point, and held there for the remainder of the freezing plateau.

The procedure for realizing the freezing plateau of the zinc cell was similar to that of the tin cell. The temperature was raised to 3 °C above the theoretical freezing plateau and allowed to stabilize overnight. To initiate the freeze, the furnace temperature was lowered to 2 °C below the freezing point at a low scan rate of 0.1 °C. A silica quartz glass tube was inserted into the reentrant well for four minutes to induce nucleation when recalescence was observed. Then the furnace temperature was raised to 0.4 °C below the freezing point. With both cells, a standard platinum resistance thermometer (Fluke – Hart Scientific model 5683 SPRT) was inserted into the cell to record the freezing plateau temperatures.

3.3 Comparison testing

The short metal-cased tin and zinc fixed-point cells were compared against a standard size fused quartz glass shell tin cell (Fluke – Hart Scientific model 5905 tin fixed-point cell) and a standard size fused quartz glass shell zinc cell (Fluke – Hart Scientific model 5906 zinc fixed-point cell) to evaluate the system's performance. Three SPRTs were used to record temperatures. The comparison testing procedure for the tin cell is described following. A similar procedure was also used for the zinc cell.

The short tin cell was placed in the furnace as shown in Figure 1(b), and the standard size fused quartz glass shell tin cell was placed in a classic fixed-point furnace (Fluke – Hart Scientific model 9114 fixed-point maintenance furnace). The freezing plateau of the short tin cell was initiated as described above in section 3.2. The freezing plateau of the standard size cell was realized according to standard procedures [3,4] 40 minutes prior to that of the short cell.

The comparison test was repeated three times for the cell. Three SPRTs were rotated between the two cells for a total of six measurements in a test. For the first test, the measurement sequence was SPRT 1 (standard cell), SPRT 2 (short cell), SPRT 3 (standard), SPRT 1 (short), SPRT 2 (standard), and SPRT 3 (short). For the second test, the sequence started with SPRT 3 (standard), SPRT 1 (short), and continued. Then for the third test, the sequence began with SPRT 2 (standard), SPRT 3 (short), etc. The purpose for rotating three SPRTs and changing the sequence for each test was to remove influences of the SPRTs' particular characteristics and differences in preheating response. Varying measurement currents of 1.0 mA, 1.414 mA, then 1.0 mA again were applied to obtain zero-power SPRT resistance values and remove self-heating effects. Before and after each test, the triple point water values R_{tpw} of the SPRTs were measured to observe stability of the probes.

4. Results and discussion

The freezing plateaus of the short tin and zinc cells are shown in Figure 3. The freezing plateau duration of the tin cell was over 10 hours before a 1.0 mK drop, and over 20 hours for the short zinc cell. Either plateau is long enough for one day's calibration work.

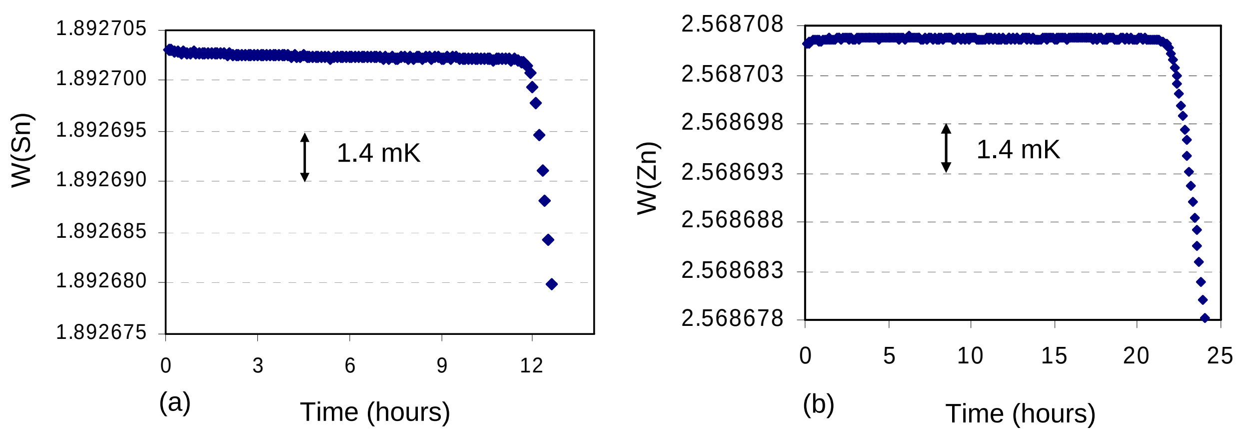


Fig. 3. Freezing plateau of the short tin cell (a) and the short zinc cell (b).

Comparison test results are shown in Figure 4 and Figure 5 for the tin and zinc cells respectively. It can be seen that the largest difference between the short tin cell and the standard size fused quartz glass shell tin cell is about 0.80 mK, and the average difference is 0.35 mK among the three SPRTs. The largest difference between the short zinc cell and the standard size fused quartz glass shell zinc cell is 0.30 mK, and the average difference is 0.05 mK for the three SPRTs.

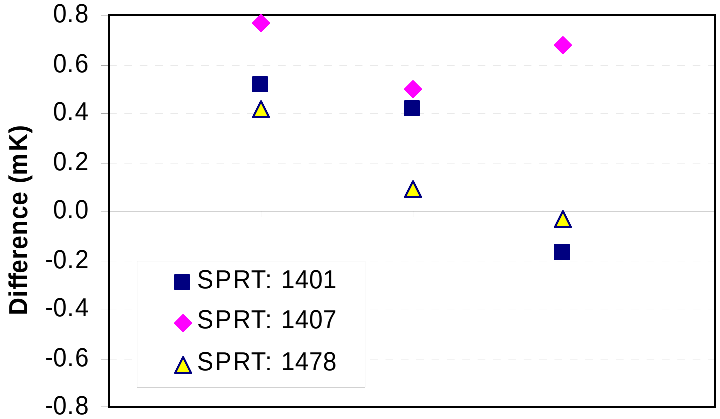


Fig. 4. Comparison between the short tin cell and standard size fused quartz glass shell tin cell.

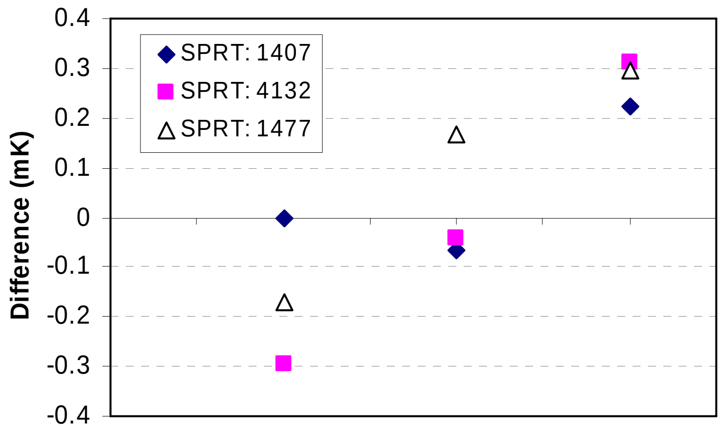


Fig. 5. Comparison between the short zinc cell and standard size fused quartz glass shell zinc cell.

From the performance of the freezing plateau and the comparison test results, it appears that the short zinc cell is more stable than the short tin cell. The freezing plateau duration of the zinc cell was double that of the short tin cell, and the difference between the short zinc cell and the standard size zinc cell was less than half of the difference between the short tin cell and the standard size tin cell. Generally speaking, the performance of a tin cell should be better than that of a zinc cell because of its lower freezing temperature. One possible reason for the observed results is because the metal purity of the zinc is higher than that of the tin. Analysis of the plateaus suggests that the purity of the zinc is over 99.99999%, and the purity of the tin is about 99.99993%. Another possible reason is related to the phase transformation characteristic during solidification of tin and zinc. More experiments are needed to further investigate this phenomenon.

5. Uncertainty estimation

The estimated uncertainties of the short tin and zinc fixed-point systems are listed in Table 1. The purities of both tin and zinc are higher than 99.9999%. The estimated expanded uncertainties (k=2) are 0.748 mK and 1.260 mK for the tin and zinc fixed-point systems, respectively. From the uncertainty analysis, it can be seen that for both tin and zinc cells, the largest uncertainty component comes from the impurities. If the purity of metal tin and zinc can be improved to 99.99999%, the expanded uncertainties can be reduced to 0.440 mK (k=2) and 0.654 mK (k=2) for the short tin and zinc cells, respectively.

Table 1. Estimated uncertainties of the short tin and zinc fixed-point systems.

Source of uncertainty	**Uncertainty Components (mK)**	
	Tin fixed-point system	Zinc fixed-point system
Resistance reading (A)	0.041	0.059
Reproducibility (A)	0.200	0.300
Total A	0.204	0.306
Impurities (B)	0.304	0.541
Hydrostatic correction (B)	0.022	0.027
Pressure correction (B)	0.010	0.013
Immersion (B)	0.040	0.040
SPRT self heating (B)	0.030	0.030
Propagated from TPW (B)	0.050	0.080
Bridge non-linearity (B)	0.020	0.029
Total B	0.314	0.551
Total standard uncertainty	0.374	0.630
Expanded uncertainty (k=2)	0.748	1.260

6. Conclusion

A new fixed-point system was developed for calibration of short (8 – 9 inches) secondary platinum resistance thermometers (PRT) and industrial platinum resistance thermometers (IPRT). The system includes specially designed short tin and zinc fixed-point cells and a maintenance furnace with a three-zone controller that provides excellent vertical temperature uniformity. The system was tested by realizing the freezing plateaus of the tin and zinc cells. Both the quality and duration of the freezing plateaus make the new fixed-point system well-suited for calibration of secondary level PRTs and IPRTs. Comparison testing results show that the mean difference between the short tin cell and a standard size fused quartz glass shell tin cell is 0.35 mK, and 0.05 mK between the short zinc cell and a standard size fused quartz glass shell zinc cell.

Acknowledgments

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