Using a Mini Triple Point of Water System to Improve Reliability in a Temperature Calibration Laboratory

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Abstract

A mini TPW system was introduced as an excellent way to measure the errors in the calibration system of a secondary level temperature laboratory. In this paper, the structure and operation of this system is briefly introduced. Also, its performance is discussed. The mini TPW system was directly compared to a traditional TPW cell. The difference between the mini TPW system and the traditional TPW cell was found to be less than 0.3 mK using an SPRT. The expanded (k=2) uncertainty of the mini TPW system is better than 0.5 mK. Several thermometers with different structures were tested in the system. Errors seen with the different thermometers between the mini TPW system and the traditional TPW system are reported.

1. Introduction

Temperature calibration laboratories not using fixed-point standards use some level of reference or standard thermometer instead, such as a standard platinum resistance thermometer (SPRT) or secondary platinum resistance thermometer (PRT), against which units under test (UUTs) are calibrated. Reference thermometers are typically sent to primary or upper level calibration laboratories for routine calibration. Often the calibration interval for a reference thermometer is one year. However, it is difficult for many users to observe and track the drift of the reference thermometer between calibrations. If the reference thermometer drifts beyond the acceptable limit during the calibration interval, the process uncertainties may be compromised requiring recall of calibration work.

Upon investigation of re-calibration results, it was found that the resistances at the triple point of water (Rtpw) of some reference thermometers were significantly different between the two calibrations, and some reference thermometers had to be removed from service due to their inadequate stability. However, the "bad" reference thermometers had been in use as standards for calibration work. No one knows when the thermometers drifted significantly.

Measurement instruments, including readouts and reference resistors, may also drift over some time period, though their drifts are usually much smaller than that of reference thermometers. It is suggested that the entire calibration system should be checked regularly.

An excellent way to measure the errors in a calibration system, including drift of the reference thermometer, is to compare against an established fixed point, such as the triple point of water.

However the cost of a typical triple point of water cell and its maintenance apparatus (as well as the time, effort, and training involved) can preclude its use in many laboratories. In order to make fixed-point standards more accessible, a mini triple point of water (TPW) system was developed a few years ago [1] [2].

To ensure that the mini TPW system is usable as a watchdog for a calibration system with many different types of reference thermometers, further research was completed. Several thermometers with different structures were tested in the system. Errors seen with the different thermometers between the mini TPW system and the traditional TPW system are reported in this paper.

2. Drifts of Reference Thermometers

Recently, we investigated a collection of SPRTs calibrated by Hart Scientific over at least four one-year calibration intervals. These SPRTs were made by various manufacturers throughout the world, and their structures vary. The investigation results are surprising—the R(tpw)s of many SPRTs drifted over 10 mK in at least one calibration interval. The R(tpw) of a few SPRTs drifted as much as 50 mK. As a sample, the drifts of the R(tpw)s of 11 metal-sheath SPRTs and eight quartz-sheath SPRTs are shown in Fig.1 and Fig. 2 respectively.

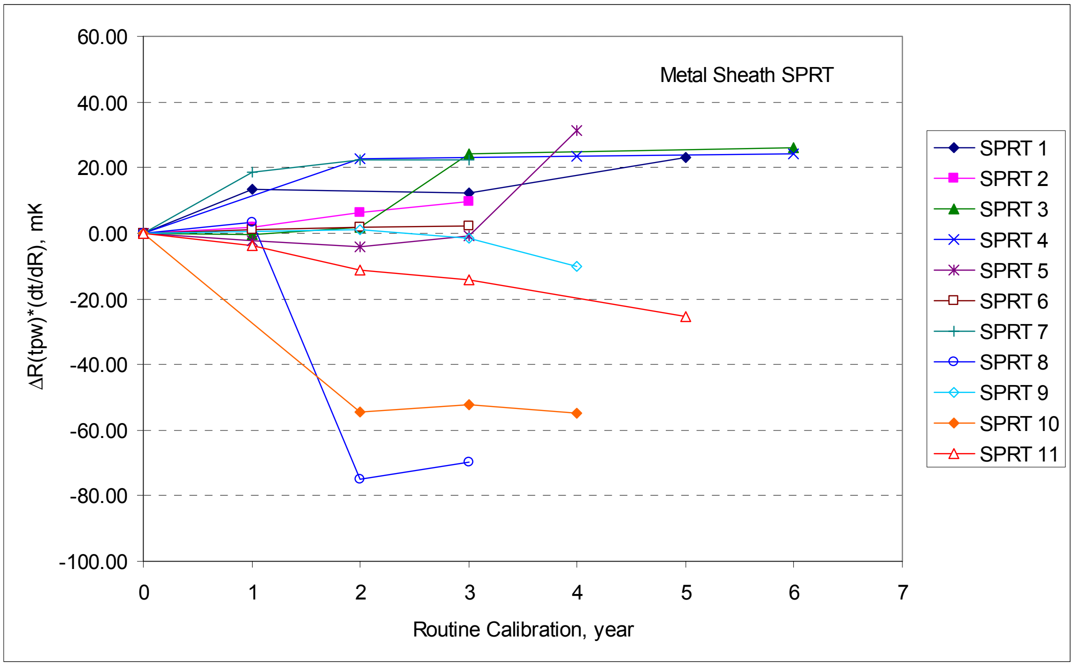

Fig. 1 The R(tpw) drifts of metal sheath SPRTs during routine calibration intervals

It was found that the R(tpw) might jump significantly in one interval, while the R(tpw) is quite stable or at least acceptable in other intervals. The R(tpw) of SPRT 8 drifted almost 80 mK between the second year and third year. The R(tpw) of SPRTs 4 and 10 drifted significantly between the first year and second year, while the same problem happened on SPRT 5 between the fourth year to fifth year. It is assumed that these SPRTs met unusual treatment during those years.

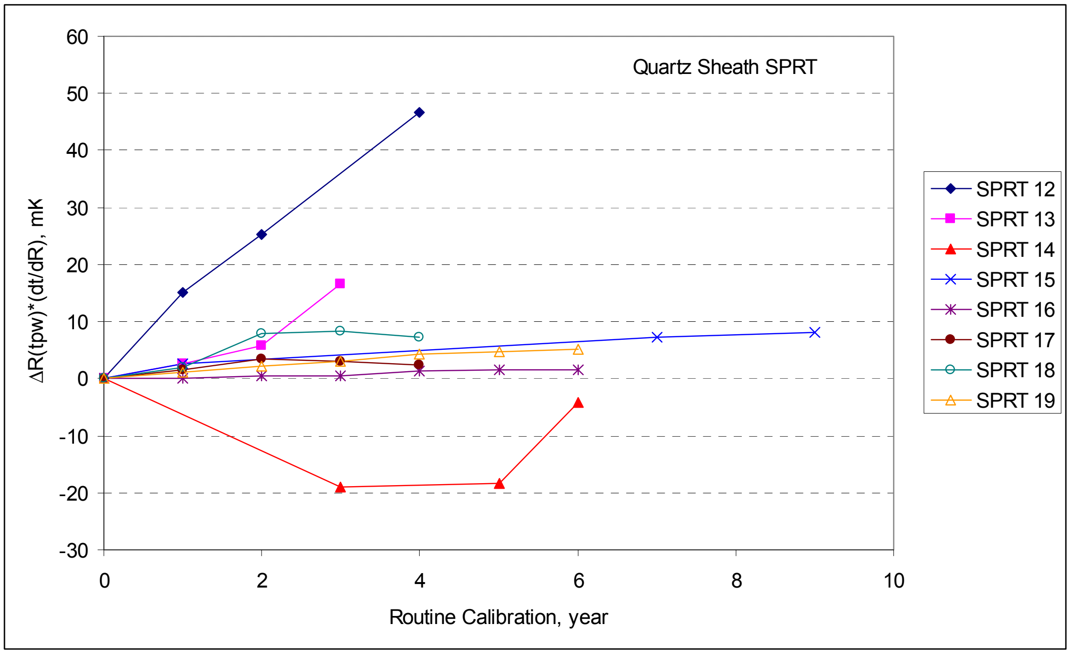

Fig. 2 The R(tpw) drifts of quartz sheath SPRTs during routine calibration intervals

Comparing Fig. 1 and Fig. 2, the long-term stability of quartz-sheath SPRTs is better than that of the metal-sheath SPRTs. The main reason, it is presumed, is that users treat the quartz-sheath SPRTs more carefully because they are expected to be more fragile. Three among eight quartz-sheath SPRTs exceeded the SPRT long-term stability limitation. The drift of the R(tpw) of a good quartz sheath SPRT should be less than 3 mK per year, depending on the usage and handling. An SPRT, whether quartz or metal sheath, should be handled very carefully to maintain its excellent stability.

During our calibrations and services for customers, it was found that some reference thermometers drifted significantly due to such various reasons as mechanical shock and overheating. Most users had not noticed the drifts until the re-calibration was completed in the upper level temperature laboratories, or because the reference thermometer did not work anymore. If the “bad” SPRT was used as reference thermometer in daily calibration, the process uncertainties may have been compromised and recalls of calibrated work may be necessary.

The reference thermometer should be checked regularly between the calibration intervals to guarantee the calibration reliability. An ideal and the easiest way to measure the errors in a calibration system, including drift of the reference thermometer, is to compare against the triple point of water.

Usually, the resistance ratio (W) of a reference platinum resistance thermometer is more stable over time than the resistance at the triple point of water. Therefore, if the R(tpw) could be updated regularly, the calibration uncertainty will improve significantly.

3. Mini TPW Cell and Realization System

The realization of the triple point of water (TPW) in traditional cells requires dry ice or liquid nitrogen to create an ice mantle and a fluid bath or ice dewar to maintain the phase equilibrium state within the cell. However, the cost of a typical triple point of water cell and its maintenance apparatus (as well as the time, effort, and training involved) can preclude its use in many laboratories. In order to make fixed-point standards more accessible, a mini triple point of water (TPW) automated system was developed a few years ago. In this paper, the system is briefly introduced again. Detailed information and performance were presented in our previous paper [1] [2].

The triple point of water can be realized automatically using a miniature TPW cell and a portable apparatus with thermo-electric cooling modules and temperature controller. The apparatus was designed with built-in programming for fast and easy operation. Three pre-set temperatures are built into the unit's controller: 5°C (melt mode), near 0°C (maintain mode), and -4°C (freeze mode). Operation is extremely simple. Enter the "freeze" mode through the front-panel buttons. When the apparatus sounds an alerts (about ten to twenty minutes later), remove the mini cell from the apparatus and give the cell a light shake. The water in the cell, supercooled at a temperature of -4°C, immediately begins to freeze. Fine needle-crystals (dendrites) of ice appear uniformly throughout the cell and approximately 5% of the water freezes in a few seconds. Return the cell into the apparatus and change the program mode to "maintain."

4. Tests, results and discussions

Measurement Apparatus

In this study, all resistances were measured using a Guildline DC automatic bridge (Model 6675). The expanded uncertainty for the measurement of the ratio of two resistances was less than 2×10^{-7}. A Leeds & Northrup 10-ohm DC standard resistor was used. The triple point of water, Hart Model 5901, with the maintenance bath, 7312, was used in the experiment.

Performance of the mini TPW system

Generally, the plateau will last from 14 to 20 hours. The temperature in a miniature TPW cell newly frozen (within the first 30 minutes) in this manner is typically about 1 mK below the triple point of water. This low initial temperature and the subsequent gradual rising in the cell's temperature are believed to be caused by structural strains in the cell's suddenly frozen ice and the subsequent relieving of this strain over time. Creating an "inner melt" around the central well (by inserting a glass rod at room temperature) can expedite this settling. The equilibrium temperature of a mini TPW cell in the above-described apparatus was compared with that of a traditional TPW cell using an SPRT. The mini TPW cell was aged for ten hours after freezing the ice before the comparison began. The average difference between the two cells was found to be within 0.1 mK. If an uncertainty less than 0.5 mK is required, it is suggested to freeze the mini TPW cell in the late afternoon and use it the next day. If an uncertainty of 1 mK is satisfactory, the cell may be used immediately after freezing the ice in the cell. Early research results were published in our previous paper [1] [2]. Recent research show that the suggestions given were conservative.

Errors introduced by inadequate aging of ice mantel

To verify the errors introduced by inadequate aging of the ice mantel, more research was recently conducted. A TPW plateau realized in the mini TPW system is shown in Fig. 3. The triple point of water realized in the mini TPW system was compared to that of the traditional TPW cell. An SPRT was measured in the traditional TPW cell before and after the SPRT was measured in the mini TPW system. The average of the two measurement results is used as the triple point of water value for the traditional TPW cell. When realizing the TPW in the mini system, the ice mantel around the reentrant well was not inner melted. At the beginning of the plateau, the triple point of water in mini system is 1 mK lower than that of traditional cell. The TPW increased from -1 mK to -0.5 mK within 2.7 hours. The difference was less than 0.3 mK after 10 hours. The plateau lasted 18 hours with a temperature difference within 0.5 mK from the traditional cell. It is suggested to allow three hours for stabilization for a desired uncertainty less than 0.5 mK with the mini TPW system.

To reduce the stabilization time, a room temperature glass tube was inserted into the mini cell for one minute as soon as the ice mantel was created. This is called the “inner melt” technique. A plateau realized in the mini TPW system with “inner melt” is shown in Fig.4. It took 1.6 hours for the TPW to stabilize within a temperature difference of 0.5 mK from the traditional TPW cell. The difference was less than 0.3 mK after 4.5 hours. However, the plateau lasted only 12 hours.

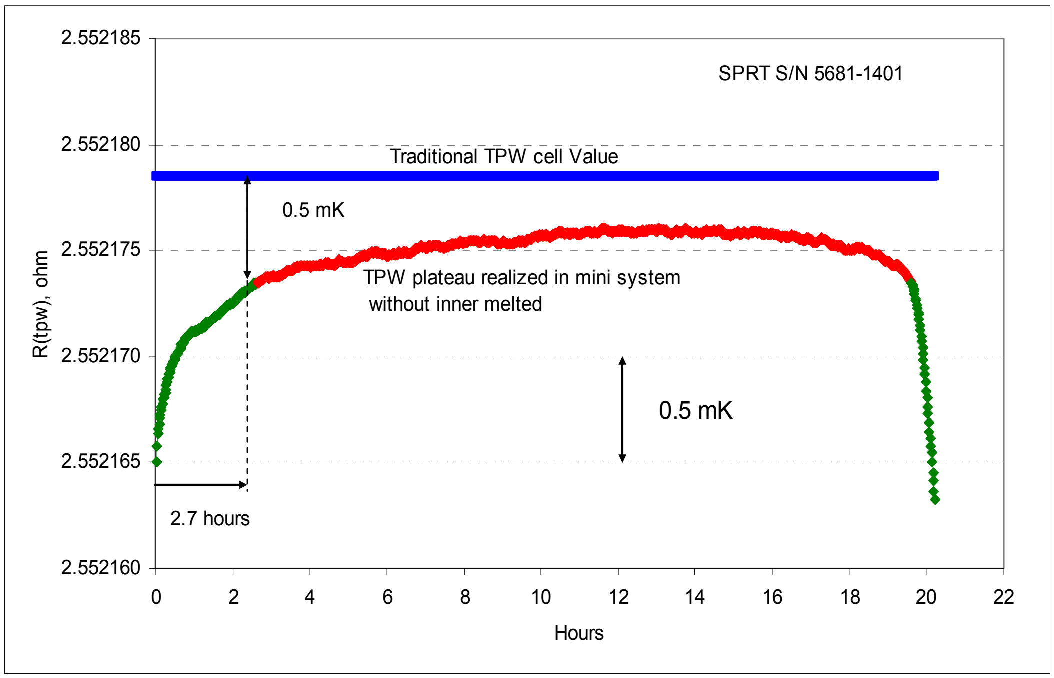

Fig. 3 TPW Plateau realized in a mini TPW system (no inner melt at the beginning)

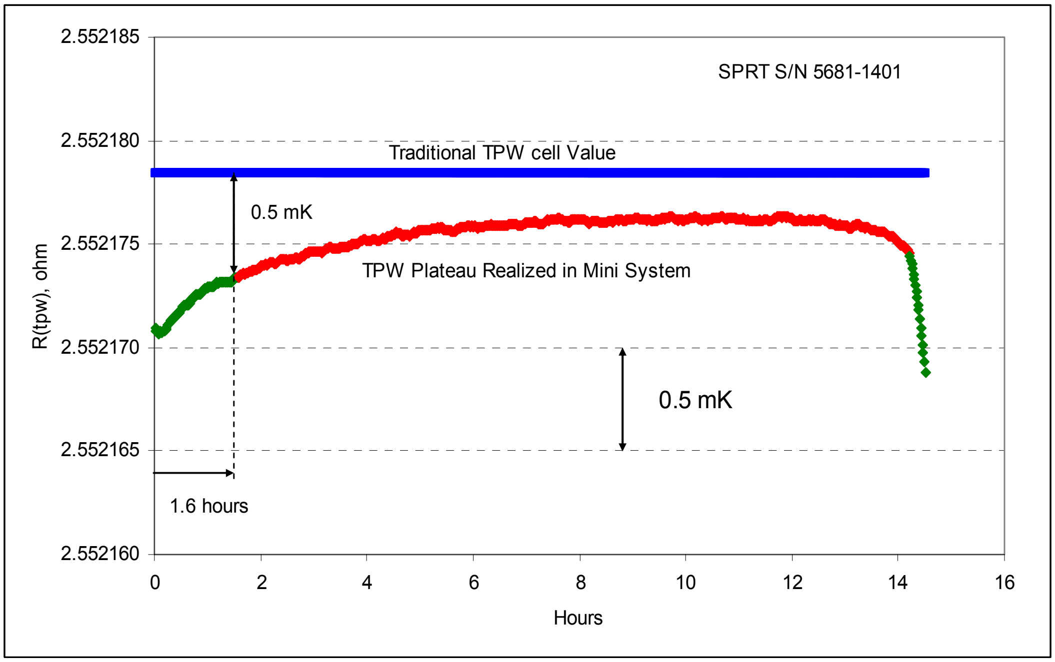

Fig. 4 TPW Plateau realized in a mini TPW system (inner melt at the beginning)

Errors introduced by different thermometer structures

As a convenient check standard tool for the temperature laboratory, the mini TPW system should be suitable for different types of reference thermometers. Since the immersion depth of the mini TPW system is 117 mm, to verify the error due to the possible inadequate immersion, many reference thermometers with different structures were tested in both a regular TPW cell and a mini TPW system. Details of the thermometers involved in the tests are listed in Table 1. The length of the longest sensor is 60mm, while the length of the shortest sensor is 5 mm. The sheath material types include Pyrex, fused silica, inconel, and alumina.

Table 1: Structure of thermometers involved in the tests

No.	Thermometer Type	sheath material	sheath diameter	sensor length	leads wire material	Leads insulation design
A	SPRT	Pyrex	7.5 mm	40 mm	Platinum	Single-bore, glass
B	SPRT	Fused Silica	7.0 mm	40 mm	Platinum	Single-bore, fused silica
C	SPRT	Fused Silica	7.0 mm	40 mm	Platinum	Capillary
D	SPRT	Fused Silica	7.0 mm	55 mm	Platinum	Single-bore, fused silica
E	SPRT	Fused Silica	7.0 mm	60 mm	Platinum	Single-bore, fused silica
F	SPRT	Inconel	5.6 mm	5 mm	Platinum	Single-bore, Alumina
G	SPRT	Inconel	5.6 mm	35 mm	Platinum	Single-bore, Alumina
H	SPRT	Inconel	6.4 mm	45 mm	Platinum	Single-bore, Alumina
I	SPRT	Inconel	5.6 mm	45 mm	Platinum	Single-bore, Alumina
J	PRT	Inconel	6.4 mm	50 mm	Platinum	Single-bore, Alumina
K	PRT	Inconel	6.4 mm	40 mm	nickel	4-bore alumina
L	PRT	Inconel	4.8 mm	35 mm	nickel	4-bore alumina
M	PRT	Alumina	6.4 mm	30 mm	Platinum	Single-bore, Alumina

The temperature difference between the mini TPW system and the traditional TPW cell using different thermometers are shown in Fig. 5. For the fused silica glass and inconel sheath thermometers, as long as the sensor lengths are no more than 55mm, the temperature errors do not exceed 0.3 mK. If the sensor length is 60 mm, the error is less than 0.5 mK. All the errors are still within normal uncertainty. However, if the sheath material is alumina, even though the sensor length is only 30 mm, the error could be 0.8 mK. Usually the sensor lengths of the commercial reference thermometers are less than 50mm. Therefore, the mini TPW system is good enough for all types of reference thermometers in secondary level temperature laboratories, including quartz sheath and metal sheath SPRTs and PRTs.

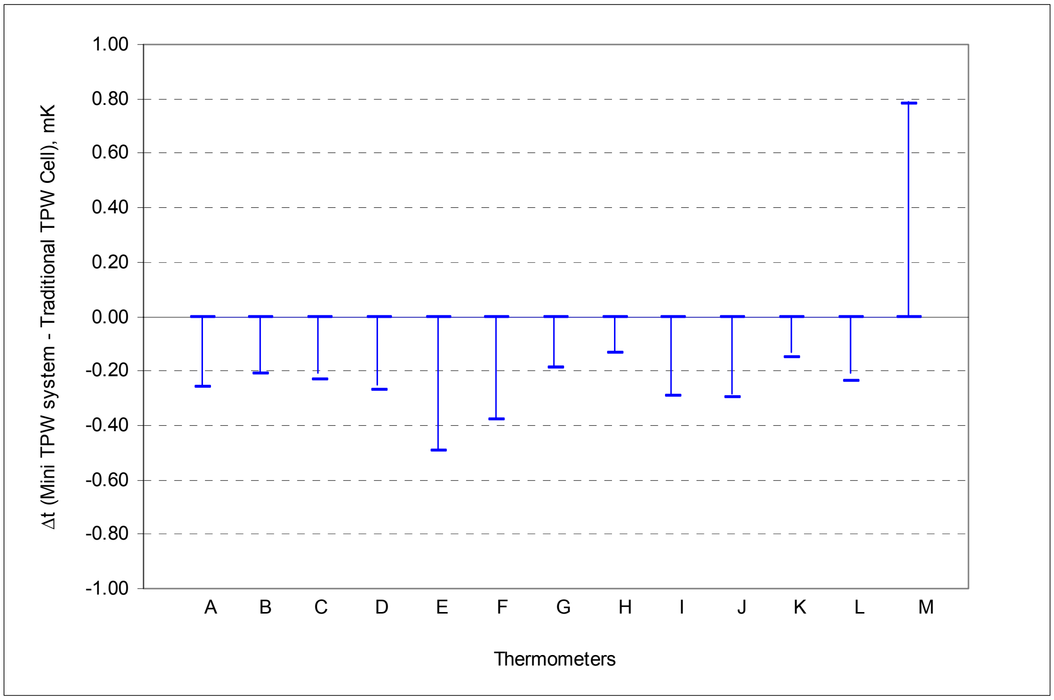

Fig. 5 Effects of thermometer structure

Check Standard of the Temperature Calibration System

Tracking the triple point of water resistance of the reference thermometer using the mini TPW system not only helps to identify drift in the reference thermometer, but it also makes drift of other components of the calibration system observable. Changes in the thermometer readout or reference resistor can also produce apparent drift of the measured R(tpw) of the reference thermometer. Other possible problems that might affect temperature measurements, such as due to environmental conditions or electrical interference, might also be made known with this valuable technique. When drift is observed, it is important to be able to identify which instrument or instruments are not stable. It can be helpful to also have reliable standard resistors for use in substitution for the SPRT or PRT to test the electrical equipment.

5. Conclusion

Investigation results show that there are many reference thermometers outside tolerance limits are still used as the standard in calibration work. Thus, the process uncertainties may have been compromised which may lead to the need to recall calibration work. To guarantee the reliability in a secondary level temperature calibration laboratory, the reference thermometer should be checked regularly. The mini TPW system is an excellent and simple apparatus to assist the laboratory in controlling their process. It allows a control chart to be maintained that indicates when a reference thermometer should be sent to an upper level temperature laboratory for re-calibration.

The difference between the mini TPW system and the traditional TPW cell was found to be less than 0.3 mK using an SPRT after the ice mantel is fully aged. The difference could be less than 0.5 mK after two or three hours of stabilizing after the ice mantel is created. The expanded (k=2) uncertainty of the mini TPW system is better than 0.5 mK. The mini TPW system is good for thermometers with sensor length no more than 60 mm.

The mini TPW system could be used as a verification and quality assurance device for a temperature calibration system and can reduce uncertainty by allowing regular updating of the resistance of the reference thermometer at the triple point of water.

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