A High-Quality Platinum Resistance Thermometer to 661°C

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Abstract

A new high quality 25-ohm platinum resistance thermometer with an inconel protection sheath was developed. The thermometer adopts SPRT design techniques and meets all of the requirements of the ITS-90. It is more durable and affordable than typical SPRTs.

The structure and design of the thermometer are briefly discussed in this paper. The test results of a group of thermometers are presented. The tests included long-term and short-term stability, thermal cycling, thermal hysteresis, insulation resistance and W(Ga) value. The test results show the thermometer to be an excellent secondary standard for a temperature laboratory in the range from –200°C to 661°C.

Introduction

Long-stem standard platinum resistance thermometers (SPRTs) are used to interpolate temperature in the range from 84K to 660°C on the International Temperature Scale of 1990 (ITS-90)[1]. They are widely used as standard or reference thermometers to calibrate other thermometers and to measure temperature precisely in primary and secondary laboratories. Most SPRTs are stable to as little as 2 mK per year at the triple point of water. However, SPRTs are fragile and expensive compared with industrial platinum resistance thermometers (IPRTs). SPRTs are easily made unstable—even damaged—after mechanical shock. For many laboratories where accuracy and stability are of foremost importance, SPRTs are the first choice, but extreme care in use is necessary to obtain the highest measurement accuracy.

For many temperature measurements and calibrations in secondary laboratories, SPRTs are more accurate than necessary and cost more than budgets allow. At the same time, typical IPRTs have not been accurate or stable enough to meet laboratory requirements. A compromise between SPRTs and IPRTs resulted in the development of a new high-quality platinum resistance thermometer (PRT).

Eight of the new PRT were developed and tested. The PRT's temperature range is from –200°C to 665°C. The thermometer adopts SPRT design techniques and meets all of the requirements of the ITS-90. For ruggedness, it also adopts some techniques used in manufacturing IPRTs. The accuracy, stability, cost, and ruggedness characteristics of the thermometer all fall between those of SPRTs and typical IPRTs. The long-term drift at the triple point of water is better than 0.02°C, and the short-term stability is better than 0.004°C. The tested thermometers have excellent thermal cycling performance, very low thermal hysteresis effects, and extremely high insulation resistance.

Thermometer Structure and Design

The structure of the thermometer is shown in Fig.1. All parts used in the thermometer are cleaned and fired carefully before assembly. The thermometer element is crafted using 0.07-mm-diameter high-purity platinum wire wound in a quartz cross support, and has a resistance at 0.01°C of 25.5 ohms. The sensor wire is first wound into a small-diameter coil. The coil is then wound on to the cross support. The coil ends are welded to the 0.3-mm-diameter lead wire. The thermometer utilizes a four-terminal construction to eliminate lead resistance. The four lead wires are fixed on the cross support. A sensor protection capsule is used to shield and hold the resistance element. A special powder mixture is filled into the sensor capsule to support the element wire to protect the element from mechanical shocks. A powder is chosen that will not contaminate the platinum and is specially mixed not only to protect the element from mechanical shocks but also to enable the platinum sensor wire to expand and contract freely. After all element parts and powder are assembled into the capsule, a pure mixture of gases, including oxygen, is filled into the sensor at high temperature. The sensor is hermetically sealed under pressure, which is adjusted to be around 101,325 Pa at the upper limit temperature (665°C).

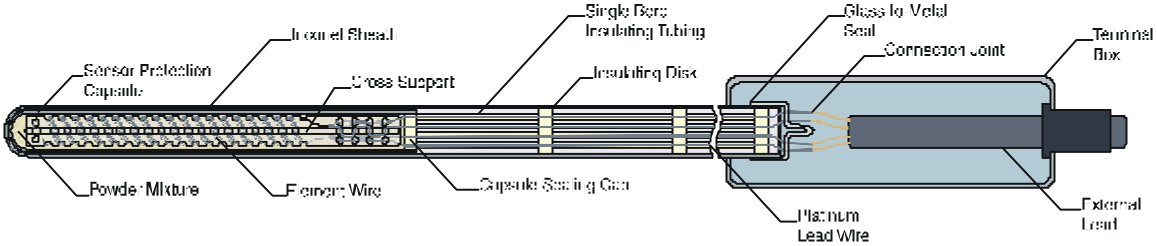


Fig. 1 Structure of the thermometer

The four thermometer leads are insulated by thin-walled single-bore alumina tubing separated by alumina disks. A 6.25-mm outside-diameter Inconel 600 sheath encases the sensor and leads in a pure gas-mixture atmosphere, which includes oxygen. The unit is hermetically sealed using a glass-to-metal seal. The junction between the platinum leads and the external copper leads is designed to minimize thermal EMFs generated by temperature differences. Four pure copper joints fixed in the thermometer's handle are used to connect the platinum leads and the external copper leads. Gold-plated spade lugs terminate the lead wires.

Testing and Results

Measurement Apparatus

The resistance of the thermometer was measured with a digital data system, including Hart Scientific's Model 1590 Super-Thermometer II. The Super-Thermometer achieves 1-ppm accuracy with an external standard resistor. A Leeds & Northrup 100-ohm DC standard resistor was used. The triple point of water and the freezing point of aluminum (two fixed points on the

ITS-90) were used in the thermometer tests. The uncertainty of the realized triple point of water was better than 0.1 mK. The freezing point of aluminum was realized using a melting curve technique through a “mini” aluminum cell and a small furnace with a 3-zone heater and a programmed controller. The uncertainty of the freezing point of aluminum was better than 5 mK.

Long-Term Stability

The long-term stability of the new high-quality PRT was tested at the triple point of water and the freezing point of aluminum after long-term annealing at 665°C for a total 1000 hours. After each 100 hours of annealing, the furnace containing the thermometers was cooled to 480°C at a rate of 3°C per minute. The thermometers were then removed from the furnace and cooled to room temperature. The resistance of the thermometers R(tp) was first measured at the triple point of water. Then the resistance of the thermometers R(Al) was measured at the freezing point of aluminum. The Rtp was then measured once again. The resistance ratio W(Al) was calculated, based on the formula W(Al)=R(Al)/R(tp).

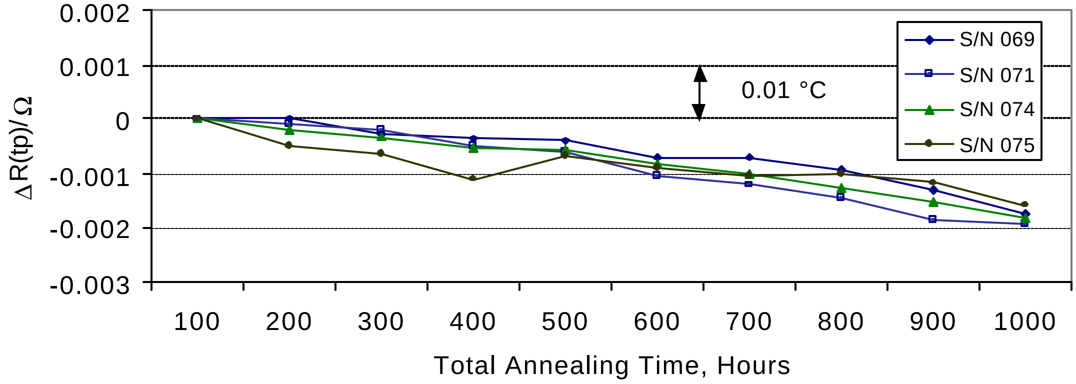


Fig. 2 Long-term stability at the triple point of water

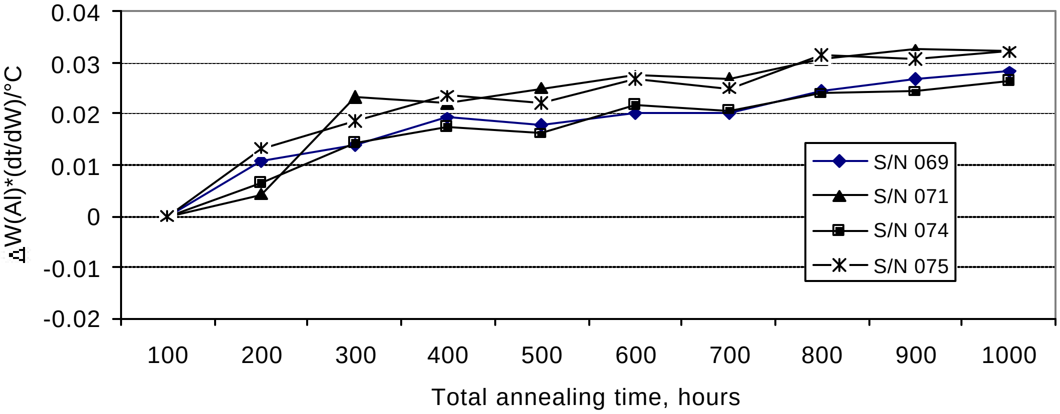


Fig. 3 Long-term stability at the freezing point of aluminum

The R(tp) and W(Al) drift for four of the thermometers, as an example, are shown in Fig. 2 and Fig. 3. The drift of R(tp) was equivalent to less than 0.02°C during the 1000 hours of annealing at 665 °C. The W(Al) went up very fast for the first 400 hours. After 400 hours, the W(Al) tended to be stable with a gradual rate of increase. For the last 600 hours, the W(Al) drift for all the test thermometers was less than 0.01°C. The fact that R(tp) went down and the W(Al) went up means that the strain on the element was eliminated gradually and the element was not contaminated by the inconel protection sheath. The test results indicate that the R(tp) and W(Al) tend to be stable. The trend of W(Al) is more pronounced than that of R(tp). This is because the resistance of the thermometers at the triple point of water includes long-term drift in addition to the elimination of strain, while the drift of the resistance ratio (usually) does not include R(tp) drift. Long-term stability tests are not yet complete and will last for years.

Three thermometers were made six months ago and were used as reference standards for testing precision dry-well calibrators manufactured at Hart Scientific. They were used heavily almost every day over the range from 50°C to 650°C. Recently, the three thermometers were re-checked at the triple point of water. The changes in the R(tp) values are listed in Table 1. In each case, the drift of R(tp) is less than 0.004°C.

Table 1. R(tp) Drift after practical use

Date	S/N 087	S/N 091	S/N 092	Notes
10/29/99	25.36905Ω	25.58697Ω	25.52565Ω	measured after production
4/4/00	25.36920Ω	25.58661Ω	25.52530Ω	provided by Hart's calibration laboratory
ΔR(tp)*(dt/dR)	0.0015°C	-0.0036°C	-0.0035°C	

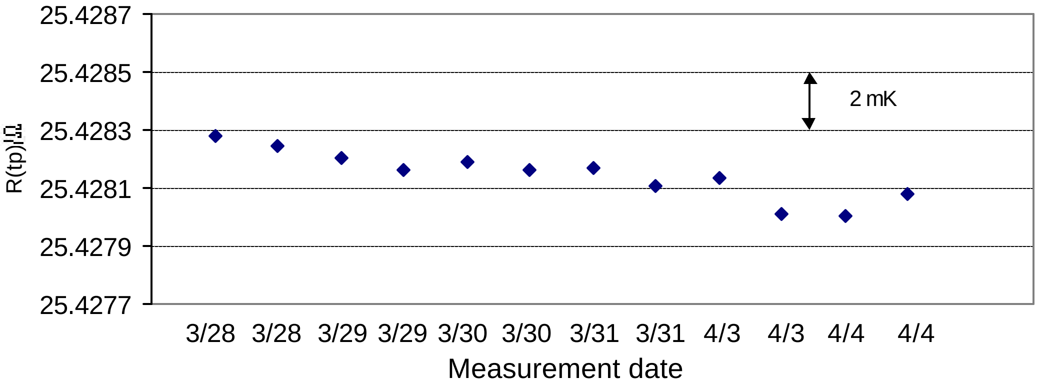


Fig. 4 Short-term stability at the triple point of water

Short-Term Stability

The short-term stability of the thermometers was also tested at the triple point of water and the freezing point of aluminum. The R(Al) and R(tp) were measured twice each day for a period of six days, and the W(Al) was calculated. During the testing, the thermometer was exposed to different temperatures from –196°C to 665°C at random intervals lasting from 10 to 60 minutes. The thermometer was always removed from the bath or furnace without annealing. As an example, the test results of one thermometer (S/N 068) are shown in Fig. 4 and Fig. 5. The test results show that the stability of R(tp) and W(Al) are better than 0.004°C and 0.005°C respectively.

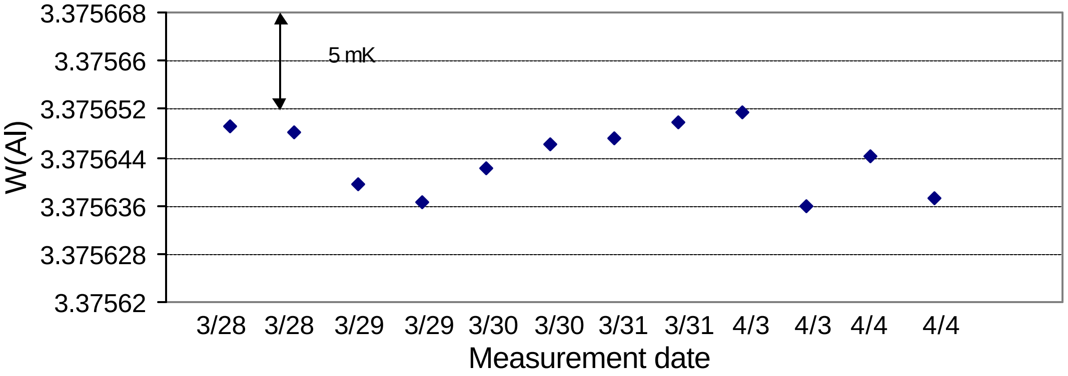


Fig. 5 Short-term stability at the freezing point of aluminum

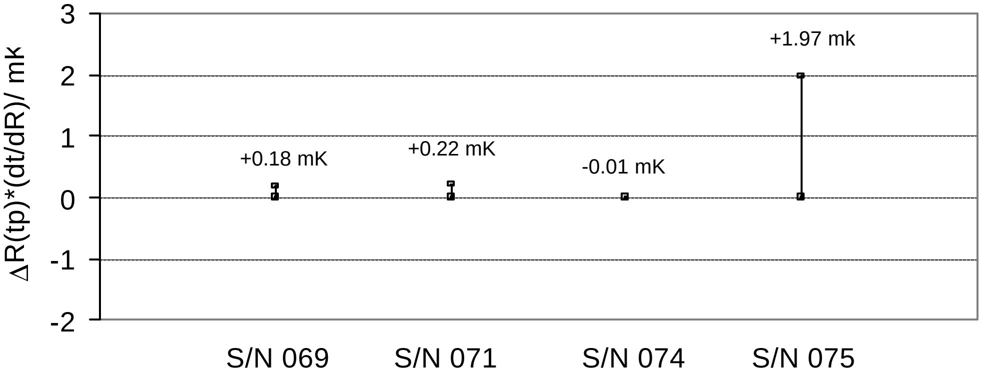


Fig. 6 R(tp) changes after thermal cycling

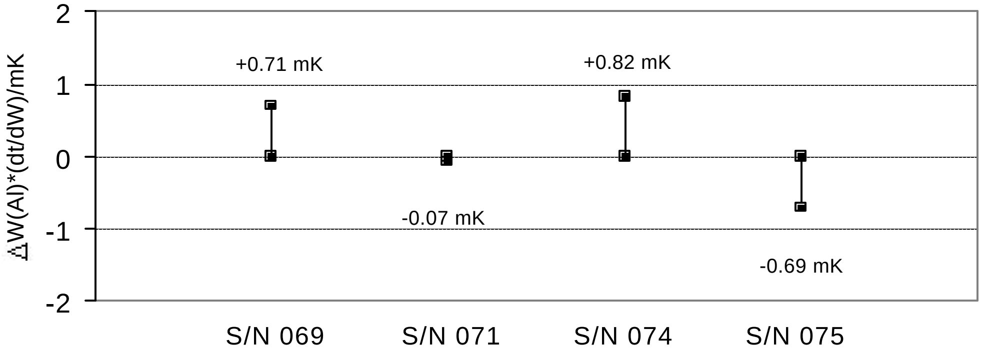


Fig. 7 W(Al) changes after thermal cycling

Thermal Cycling Effects

Cooling a thermometer rapidly from high temperatures to room temperature can quench excess lattice-site vacancies in the platinum crystal structure causing the R(tp) of the thermometer to increase[2]. For SPRTs used at temperatures above 450°C but less than 700°C, the thermometers should first be annealed for two hours at the maximum operating temperature[3]. Afterward, the furnace containing the thermometers should be cooled to 450°C with a cooling rate of 2°C per minute, then the thermometers could be removed from the furnace to room temperature.

Because the new PRT is a secondary standard, the annealing procedure should be simplified for convenience. Six thermometers were tested to investigate the effects of the thermal cycling (without annealing) from –196°C to 665°C. The test procedure was as follows:

1. The resistance of the thermometers was measured at the freezing point of aluminum and the triple point of water before thermal cycling.
2. The thermometers were immersed into a Liquid Nitrogen (LN_2) bath (-196°C) for 10 minutes.
3. They were removed from the bath and warmed to room temperature.
4. They were inserted into a furnace with a temperature of 665°C for 10 minutes.
5. The thermometers were taken out of the furnace and cooled to room temperature.

This is one thermal cycle. The same procedure was repeated ten times. After ten thermal cycles without annealing, the resistance of the thermometers at the triple point of water and the freezing point of aluminum was again measured. The comparison results for R(tp) and W(Al) before and after the thermal cycling are shown in Fig. 6 and Fig. 7. The differences of R(tp) and W(Al) before and after thermal cycling are less than 2 mK and 1 mK respectively. This indicates that the new PRTs show excellent thermal cycling effects. The annealing procedure is not necessary for them as a secondary standard, unless the thermometer has experienced mechanical shock.

Thermal Hysteresis Effects

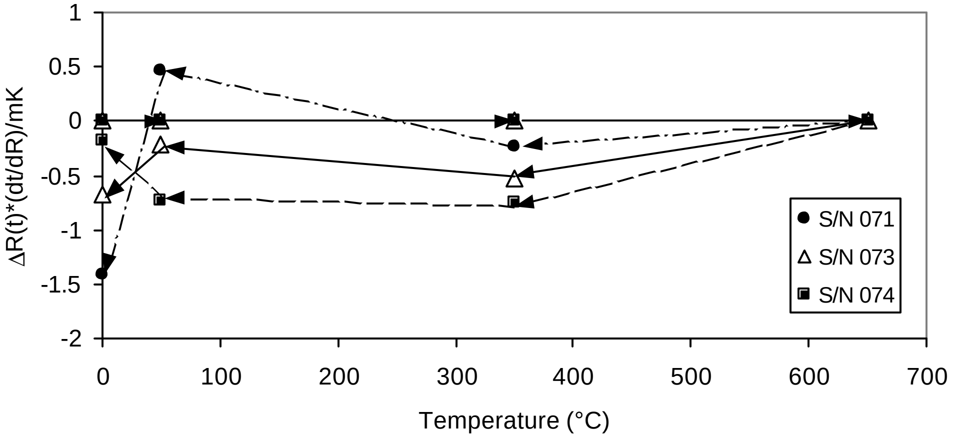
The nature and evaluation methods of thermal hysteresis in platinum resistance thermometers have been presented by D. J. Curtis[4]. In order to investigate whether the new PRTs are susceptible to thermal hysteresis, we calibrated the thermometers by comparison with an SPRT at the triple point of water and at three other points over the temperature range from 0.01°C to 650°C for both increasing and decreasing temperatures. Three thermometers were first calibrated at the triple point of water. Then they were calibrated by comparison at 50°C, 350°C, 650°C, 350°C and 50°C in that order. During the comparison, the thermometers were not moved. After comparison, the thermometers were calibrated at the triple point of water again. The data measured during the half-cycle of increasing temperatures is compared with the data measured during the half-cycle of decreasing temperatures and is shown in Fig. 8.

To avoid instrumentation errors during the comparison, the same SPRT and the same two resistance measuring instruments (Hart 1590 Super-Thermometers) were used throughout the test. Each of the test thermometers used the same Super-Thermometer (through a scanner), while the SPRT used a different Super-Thermometer. Each of the test thermometers was compared with the SPRT, one at a time, at each temperature point. During each comparison, data from the SPRT and one of the test thermometers were simultaneously recorded for 5 minutes. This procedure eliminated drift effects resulting from the temperature controller. The system error between the two Super-Thermometers is not introduced into the comparison results, because the comparison was done between the same thermometers using the same Super-Thermometer at the same temperature point. Each temperature point was measured twice during each temperature cycle. During the comparison, a furnace with a temperature range from 30°C to 680°C was used.

The horizontal and vertical temperature gradients of the furnace could be partially eliminated because they were stable at each temperature point. The accuracy of this comparison system was better than 5 mK.

The test results show no detectable hysteresis for the new PRTs. The resistance differences were equivalent to less than 2 mK. These differences are within the range of short-term stability and comparison accuracy.

Fig. 8 The test results of thermal hysteresis



Insulation Resistance

It is evident that electrical insulation leakage is a significant source of thermometer error at temperatures above 500°C and can increase rapidly with thermometer stem temperatures [5]. The insulation resistance between the four leads and the Inconel sheath can be easily measured on any thermometer. However, it is difficult to check the insulation resistance across the sensing element. In order to do so at high temperatures, a special thermometer was constructed. This thermometer was virtually almost identical to the new thermometer in every respect, except the element wire, which was cut at its midpoint. This offered a more reliable way to measure the insulation leakage between the two pairs of current-potential leads. The insulation resistance was measured under 100 volts by the Megohmmeter (AEMC Instruments Model 1000N). The measurement results are listed in Table 2. Based on the measurement method suggested by ASTM[6], the readings should be taken within 10 seconds of the voltage application. The measurements across the leads were taken with both normal and reversed polarity, because the insulation resistance changes after reversing polarity. The low reading measured, which is 90 MΩ at 665°C, should be used to calculate the error caused by electrical insulation leakage. Therefore, the error due to electrical insulation leakage is about 0.8 mK at 665 °C.

Table 2 Insulation resistance for S/N 0107 PRT

	22 °C	665 °C
Leads to sheath	40 GΩ	100 MΩ
Leads to leads	7 GΩ	90 MΩ
Leads to leads (reverse)	----	150 MΩ

W(Ga) Value

For a platinum resistance thermometer to be acceptable as an ITS-90 interpolating instrument, it must be made from pure platinum and exhibit a resistance ratio at the gallium melting point W(Ga) larger than 1.11807[1]. Six thermometers were calibrated at the melting point of gallium. The W(Ga) was calculated and is listed in Table 3.

From the W(Ga) value, we can also determine whether the thermometer was contaminated after it was used at high temperature. Before being calibrated at the melting point of gallium, these test thermometers were annealed for more than 1000 hours and underwent various tests such as those described in this paper. The W(Ga) remained very high, which means the thermometers were not contaminated. It is the contamination from PRTs' metal sheaths that restricts most PRT manufacturers from raising their thermometer's' upper temperature limits. Typically, a PRT with a metal sheath can only be used practically up to 500°C.

Table 4. The resistance ratio at the melting point of Gallium

	S/N 069	S/N 070	S/N 071	S/N 072	S/N 074	S/N 075
W(Ga)	1.1181193	1.1181192	1.1181195	1.1181161	1.1181206	1.1181152

Conclusions

The new PRT was developed to fill the gap between highly accurate but fragile SPRTs and rugged but less accurate IPRTs, based on a compromise of structure design. The structure of the thermometer is similar to that of an SPRT. The differences include the application of certain techniques to protect the element from contamination at high temperature and mechanical shocks. It is important to find just the right material for the sensor. Not only does the sensor need protection from contamination and mechanical shock, it should also exhibit excellent performance in the areas of thermal cycling and thermal hysteresis. The test results indicate that the new high-quality platinum resistance thermometer is an excellent temperature standard for secondary laboratories in the temperature range from –200°C to 661°C. The new thermometer is shown to have significant potential for secondary temperature laboratories, which typically need accuracy and stability of 0.02°C at the triple of water.

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