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Comparison System for the Calibration of Capsule-Type Standard Platinum Resistance Thermometers at NMIJ/AIST

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Abstract A new comparison system has been constructed using a Gifford-McMahon type cryogenic refrigerator for the calibration of capsule-type standard platinum resistance thermometers (CSPRTs) below 273.16 K at the National Metrology Institute of Japan (NMIJ). The system can compare six CSPRTs at once. A gold-plated comparison block, in which CSPRTs are mounted for calibration, is made from oxygen-free high-conductivity copper. The standard uncertainties related to the temperature control of the system are estimated to be 0.04 mK. The calibrated values for CSPRTs and a rhodium–iron resistance thermometer obtained using the comparison system are in good agreement with those obtained by the direct realization of the low-temperature fixed points of the ITS-90 within the combined standard uncertainty for the calibration using the comparison system.

Keywords Calibration · ITS-90 · Low temperature · Platinum resistance thermometer · Uncertainty

1 Introduction

In the region between 13.8033 K and 273.16 K, the International Temperature Scale of 1990 (ITS-90) is defined by capsule-type standard platinum resistance thermometers (CSPRTs) calibrated at certain defining fixed points, i.e., the triple points of H_2O (273.16 K), Hg (234.3156 K), Ar (83.8058 K), O_2 (54.3584 K), Ne (24.5561 K), and equilibrium H_2 (13.8033 K), and two other points near 17.0 and 20.3 K realized by equilibrium H_2 vapor pressure or an interpolating constant-volume gas thermometer [1].

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The methods of realizing these low-temperature fixed points involve complex experimental apparatus and procedures, and the calibration of CSPRTs by realizing the fixed points consumes significant time.

Previous work at the National Research Laboratory of Metrology (now integrated into the National Metrology Institute of Japan (NMIJ)/AIST) involved the development of a comparison system for low-temperature capsule-type resistance thermometers using a Gifford-McMahon (GM) type closed-cycle cryogenic refrigerator [2]. The advantages of closed-cycle cryogenic refrigerators include easy handling without any liquid cryogens such as liquid He and liquid N_2, easy operation, and a nearly maintenance-free system. Thus, recently, closed-cycle cryogenic refrigerators have been used for the calibration of low-temperature capsule-type resistance thermometers [2–10]. Furthermore, the time and cost of calibrating CSPRTs can be reduced using a comparison system that employs a closed-cycle cryogenic refrigerator in comparison to the time and cost for the direct realization of the low-temperature fixed points of the ITS-90.

However, our previous comparison system showed a large drift of temperature, about $1\,\mathrm{mK \cdot h^{-1}}$ below 20 K, that dominated the uncertainty of the calibrations [2]. The large temperature drift may be caused by a lack of sensitivity of the temperature sensor controlling the copper comparison block in which the thermometers to be calibrated and a reference thermometer are mounted. Since the copper comparison block in the previous system is directly linked to the cold head of a GM refrigerator via copper wires and leads for resistance measurement, temperature fluctuations of the cold head may also affect the control of the temperature of the copper comparison block.

We have constructed a new comparison system for the calibration of CSPRTs at NMIJ using a GM refrigerator with improved temperature stability of the copper comparison block. It has been confirmed that the temperature of the comparison block is stable within 0.06 mK at temperatures near all the low-temperature fixed points needed for CSPRTS, and the standard uncertainties related to the temperature control of the comparison system are estimated to be less than 0.05 mK. First, we will describe the results related to the temperature control of our comparison system. We have also checked the performance of the comparison system by investigating the differences between calibration values obtained using the comparison system and those obtained by realizing the low-temperature fixed points directly. Secondly, we will demonstrate the results of the investigation at each low-temperature fixed point.

2 Experimental

Figure 1 shows a schematic side view of our new comparison cryostat for the calibration of CSPRTs. The cryostat contains a detachable, gold-plated comparison block made from oxygen-free high-conductivity copper. It is cylindrically symmetric and has six wells for CSPRTs in the annular region. The CSPRTs are liberally coated with vacuum grease during insertion into the wells to enhance their thermal contact with the block. Electrical leads are thermally anchored at several temperature zones of the cryostat before a final thermal anchoring around the outside of the comparison block.

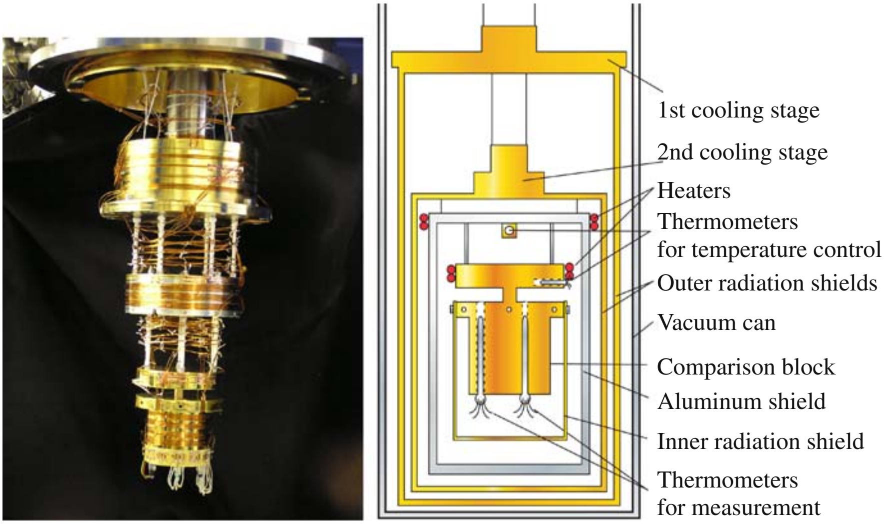

Fig. 1 Photograph and schematic side view of the NMIJ comparison cryostat

The lower part of the comparison block is completely surrounded by an inner radiation shield to maintain a uniform thermal condition and to avoid the effect of radiation from the outside. The comparison block is also enclosed in an isothermal aluminum radiation shield, which the previous cryostat does not have. The isothermal aluminum shield is completely covered by outer radiation shields fixed to the two cooling stages of the cryostat.

The cryostat has a commercial anti-vibration unit to suppress mechanical vibration due to the cold head. The anti-vibration unit separates the mechanical vibration of the refrigerator from the cryostat by using a bellows. The effect of the vibration is nominally reduced to about 5% or less by using the anti-vibration unit.

The temperature of the comparison block is stabilized by a PID controller, which controls an electric heater on the comparison block. The controller measures the temperature by using an AC resistance bridge with a ceramic-encapsulated rhodium–iron resistance thermometer (RIRT). The temperature of the isothermal aluminum shield is controlled just below the comparison block temperature using a temperature controller and a ceramic-encapsulated RIRT. The temperature difference between the comparison block and the isothermal shield is about 0.2 K, and the typical heater power of the comparison block is less than 0.5 mW. The cooling power of the 2nd cooling stage is controlled at each calibration temperature to reduce the heater power to the isothermal aluminum shield to less than 40 mW. The typical heater power applied to the isothermal shield is 20 mW.

CSPRTs and/or RIRTs are calibrated by comparing them with a reference thermometer, which is of the same type as the CSPRT or RIRT and calibrated according to the ITS-90 definition. Usually, at least one check thermometer of the same type, and calibrated according to the ITS-90, is used during the comparison calibration. To avoid the instability of thermometers influencing the estimate of the ability of the comparison system, we selected and used stable CSPRTs and RIRTs for this study. For example, the stability of the CSPRTs at the triple point of water is better than 0.1 mK over several years and the stability of the RIRTs at the triple point of Ne is also better than 0.1 mK over several years.

To assess the performance of the comparison system, we investigated the differences between calibrations of CSPRTs obtained using the comparison system and those obtained by realizing the triple points of Hg, Ar, O_2, and Ne at each fixed-point temperature. We also examined the differences between calibrated values of RIRTs obtained using the comparison system and those obtained by realizing the triple point of e-H_2 and using an interpolating constant-volume gas thermometer near 17 and 20.3 K. The results of the realization of these low-temperature calibration points for CSPRTs were reported in [11] and [12].

The resistances of the CSPRTs and RIRTs are measured using an automatic direct-current comparator bridge. Resistance standards used for this study are traceable to the national resistance standards (the quantized Hall resistance) via calibrations by the Electricity and Magnetic Division of NMIJ. The stability of the resistance standards was confirmed to be within 0.1 ppm by periodic calibrations spanning several years. The excitation current, I, was 0.5 mA for measurements of the RIRTs. For measurements of the CSPRTs, the excitation currents were $I = 2$ mA at the triple point of Ne and $I = 1$ mA at the triple points of O_2, Ar, and Hg. The calibration values are given for zero thermometer current by extrapolating the observations made at I and $\sqrt{2}I$ to correct for self-heating effects.

3 Results and Discussion

3.1 Temperature Control of the Comparison System

Figure 2 shows an example of the temperature stability of the comparison block at temperatures near the low-temperature fixed points of the ITS-90. The temperature of the comparison block is stable within 0.08 mK at these temperatures. Our previous comparison system showed a large drift of temperature of about $1\,\mathrm{mK \cdot h^{-1}}$ below 20 K, which may be caused by the lack of the sensitivity of the small platinum resistance thermometer used as the temperature sensor of the controller for the copper comparison block [2]. Since the sensitivity of the RIRT used as the temperature sensor for the new system is better than that of the platinum resistance thermometer at low temperature, the temperature stability of the comparison block is improved, as shown in Fig. 2. The isothermal radiation shield, which the previous cryostat does not have, between the comparison block and the 2nd cooling stage is also effective in obtaining better temperature stability of the comparison block with the new system.

On the assumption that the temperature stability is described by a symmetric, rectangular *a priori* probability distribution, the standard uncertainty, $a/\sqrt{3}$, is calculated, where a is half of the estimated width of the distribution. Thus, the standard uncertainty due to the temperature instability is estimated to be 0.023 mK.

The temperature uniformity of the copper comparison block due to heat flow into the block and its thermal gradient is estimated by changing the temperature difference

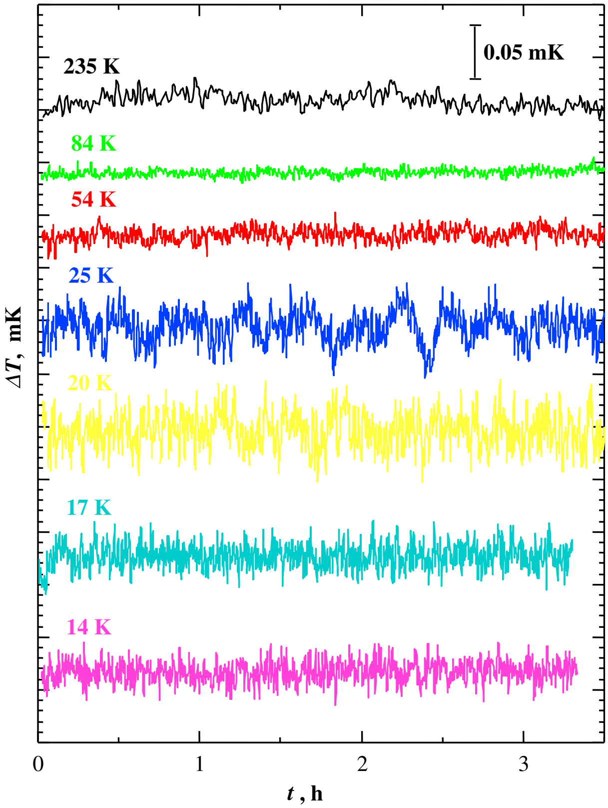

Fig. 2 Temperature stability of the copper comparison block

between the isothermal aluminum radiation shield and the copper comparison block and varying the heater power on the copper comparison block in the same manner as reported earlier [2]. Figure 3 shows typical results of the temperature shift plotted against the heater power on the copper comparison block at temperatures near the triple points of O_2 and Ne. The apparent temperature of the CSPRTs changes with increasing heater power, whereas the ceramic-encapsulated RIRT used for the temperature control of the copper comparison block is maintained at the same temperature (i.e., same resistance). Since the effect of the heater power on the copper comparison block is different at each fixed-point temperature, we always monitor the heater power to the copper comparison block to ensure that the temperature uniformity of the comparison block is below 0.06 mK during the calibration of CSPRTs. The heater powers to the comparison block are typically less than 0.5 and 0.3 mW for the triple points of O_2 and Ne for this study, respectively. On the assumption that the effect of the temperature shift is described by a symmetric, rectangular *a priori* probability distribution, the standard uncertainty due to the temperature gradient is estimated to be 0.035 mK.

The standard uncertainty component of the comparison related to the temperature control of our comparison system will be estimated by combining the above two

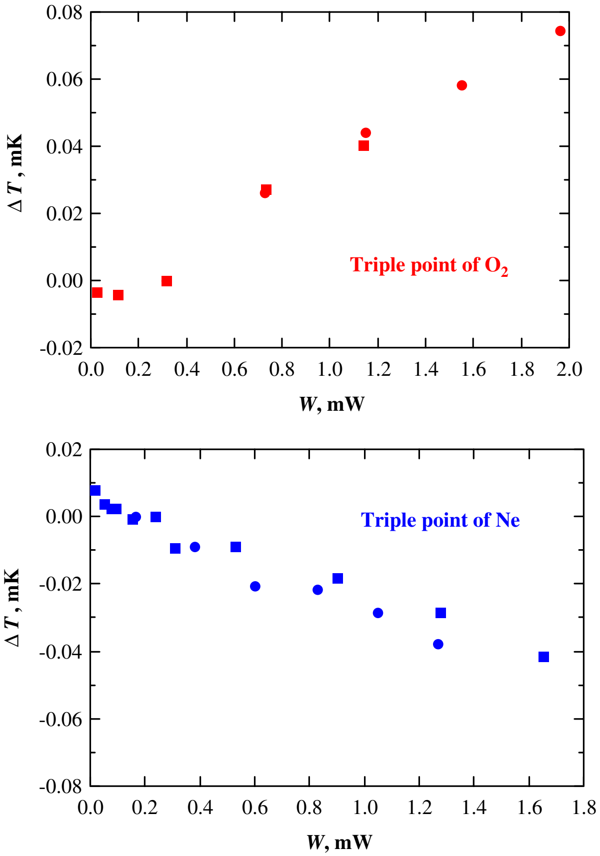

Fig. 3 Examples of temperature shift, ΔT, of standard platinum resistance thermometers in the comparison block caused by changing the temperature difference between the aluminum shield and the comparison block near the temperature of the triple points of O_2 and Ne. The horizontal axis is the heater power, W, of the electric heater on the comparison block required to maintain a certain temperature for the ceramic-encapsulated rhodium-iron resistance thermometer inserted in the comparison block after changing the temperature difference between the isothermal aluminum shield and the comparison block

standard uncertainties. Thus, the standard uncertainty of the comparison is 0.04 mK, which is much less than the combined standard uncertainty of the realization of the low-temperature fixed points at NMIJ [11].

3.2 Performance of the Comparison System for the Calibration of SPRTs

To check the performance of the comparison system, we have investigated the difference between calibration values of CSPRTs obtained with the comparison system and those obtained by realizing the triple points of Hg, Ar, O_2, and Ne [11] at each fixed point. We have also compared calibration values of a RIRT obtained using the comparison system with those obtained by realizing the triple point of e-H_2 [11] as

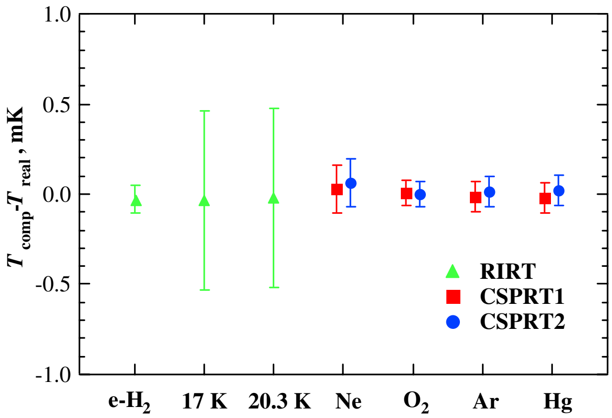

Fig. 4 Difference between the calibration values for capsule-type standard platinum resistance thermometers (CSPRTs) and a rhodium–iron resistance thermometer (RIRT) obtained by the comparison system (T_{comp}) and those obtained by the direct realization of low-temperature fixed points of the ITS-90 (T_{real}) [11,12]. Error bars are the combined standard uncertainties for the calibration using the comparison system (Table 1)

well as with those obtained near 17.035 and 20.27 K using an interpolating constant volume gas thermometer [12] at NMIJ/AIST.

Figure 4 shows the difference between the calibration values obtained with the comparison system and those obtained by the direct realization of the low-temperature fixed points. The error bars represent the combined standard uncertainty for the calibration using the comparison system (see Table 1). The calibrated values of CSPRTs and RIRTs obtained with the comparison system are in good agreement with those obtained by the direct realization of the low-temperature fixed points of the ITS-90 within the combined standard uncertainty. Figure 5 shows the repeatability of

Table 1 Uncertainty budgets for the calibration of a capsule-type platinum resistance thermometer at low-temperature fixed points using the comparison system

Fixed point Component uncertainty (mK)	e-H_2[a]	17 K	20.3 K	Ne	O_2	Ar	Hg
Temperature control	0.04	0.04	0.04	0.04	0.04	0.04	0.04
Bridge accuracy	0.04	0.02	0.01	0.01	0.01	0.01	0.01
Standard resistor	0.02	0.02	0.02	0.01	0.01	0.02	0.02
Self-heating	0.01	0.01	0.01	0.01	0.01	0.01	0.01
Reference scale[b]	0.07	0.49	0.49	0.14	0.03	0.07	0.16
Ref. therm. measurements	0.03	0.03	0.03	0.05	0.05	0.06	0.07
Temperature correction	0.02	0.01	0.01	0.02	0.01	0.01	0.03
Standard combined uncertainty (mK)	0.10	0.49	0.49	0.16	0.07	0.10	0.17

[a] Uncertainty in the case of isotopic correction based on the technical annex for the ITS-90 [13]
[b] Realization of the low-temperature fixed points has been reported elsewhere [11,12]

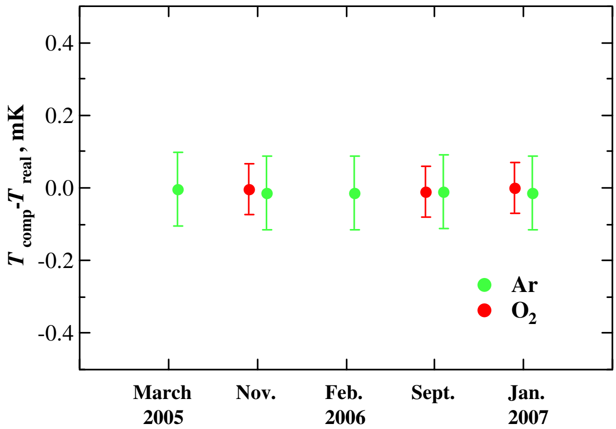

Fig. 5 Reproducibility of the calibration for a standard platinum resistance thermometer using the comparison system at the triple points of Ar and O_2. Vertical axis, $T_{comp} - T_{real}$, is the difference between the calibration values of the capsule-type standard platinum resistance thermometer (T_{comp}) and those obtained by the direct realization of low-temperature fixed points of the ITS-90 (T_{real}) [11,12]. Error bars are the combined standard uncertainties for the calibration using the comparison system (Table 1)

the calibration values of a SPRT obtained using the comparison system at the triple points of Ar and O_2. The data confirm that the calibration values are sufficiently stable over a period of more than 1 year. These results indicate that our new comparison system has sufficient performance for the calibration of CSPRTs in the low-temperature region.

4 Conclusion

We have constructed a new comparison system for the calibration of capsule-type standard resistance thermometers (CSPRTs) at the National Metrology Institute of Japan (NMIJ) using a GM refrigerator. It has been confirmed that the temperature stability of the copper comparison block is improved in comparison to that of our previous system [2]. The standard uncertainty of the comparison that is related to the temperature control of the comparison system is estimated to be 0.04 mK, which is much less than the combined standard uncertainty of the realization of the low-temperature fixed points at NMIJ [11].

The calibrated values of CSPRTs and a rhodium–iron resistance thermometer obtained using the comparison system are in good agreement with those obtained by the direct realization of the low-temperature fixed points of the ITS-90 within the combined standard uncertainty for the calibration using the comparison system. These results indicate that our new comparison system has sufficient performance for the calibration of CSPRTs in the low-temperature region.

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