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LOW TEMPERATURE CALIBRATION FACILITIES AT KRISS

Inseok Yang, Yong-Gyoo Kim, Chang Ho Song, Kee Hoon Kang and Kee Sool Gam

Korea Research Institute of Standards and Science, Daejeon, Korea, dragon@kriss.re.kr

Abstract – We review the low temperature calibration facilities at the Korea Research Institute of Standards and Science. For the calibration of capsule-type standard resistance thermometers, open and sealed cells of cryogenic fixed points and their realization system were manufactured and used. A sealed cell of the triple point of Argon for long-stem standard platinum resistance thermometers was also manufactured. For calibration by comparison, a controllable comparison cryostat and a nitrogen boiling point temperature block were made. We also review our traceability scheme and supporting international key comparisons for the dissemination of low temperature standards.

Keywords: low temperature, cryogenic fixed point, calibration

1. INTRODUCTION

Temperature standards using standard platinum resistance thermometers (SPRTs) below the triple point (TP) of mercury (–38.8344 K) are defined by the TPs of pure gas elements, such as argon (Ar), oxygen (O_2), neon (Ne) and equilibrium hydrogen (e-H_2) [1]. In this report, we provide a comprehensive review of the current status of the low temperature calibration facilities at the Korea Research Institute of Standards and Science (KRISS) and our mid-term goals for improving the facilities. Different realizations of the fixed points from open cryogenic cells, sealed-type cells, and argon sealed cells for long-stem SPRTs are reviewed. Furthermore, comparison calibration facilities, including temperature-controllable cryostats and liquid nitrogen boiling point temperature blocks are reviewed.

2. LOW TEMPERATURE FACILITIES

2.1. Open cells

In 1997, two capsule-type SPRTs were calibrated at the open cells of the TP of Ne, O_2 and Ar, and their realization systems. The two SPRTs were used in the international key comparison of capsule-type SPRTs from 13.8 K to 273.16 K [2]. The measurement of the TP of e-H_2 had not been completed at the time and was therefore not submitted. The claimed uncertainty (k = 1) for the TP of Ne was 0.18 mK and 0.14 mK for both O_2 and Ar. The difference between the result of the KRISS measurement and the Key Comparison Reference Value was less than the combined uncertainty for the comparison, with an exception for one SPRT at the TP of Ar. The system for the open cells has not been operated since the key comparison. We plan to resume operation of this system, including the realization of the TP of e-H_2.

2.2. Sealed cells

The sealed cells of e-H_2, Ne, O_2 and Ar were manufactured by IMGC (Istituto di Metrologia 'Gustavo Colonnetti') in 1999, and the cryostat for the realization of the sealed cells, as shown in Fig. 1, was used for the realization. The manufacturer claimed that the uncertainties (k = 2) of the temperature realized by these cells were 0.25 mK for e-H_2, and 0.2 mK for the others. The cryostat can be cooled down to ~17 K without the use of liquid helium. However, additional cooling by liquid helium is necessary for the TP of e-H_2. So far, the TPs of Ne, O_2 and Ar have been used for the calibration of capsule-type SPRTs. Two silicon diode thermometers (Diode1 and Diode2) monitor the temperature at the two outer shields (Shield1 and Shield2), and the temperature of the inner shield (Shield3) is controlled by the closed control loop using Heater2 and thermometer, Diode3. To realize the adiabatic melting temperature of the TP, the temperature of the inner shield is controlled to a temperature near the TP and heat pulses are applied by Heater1.

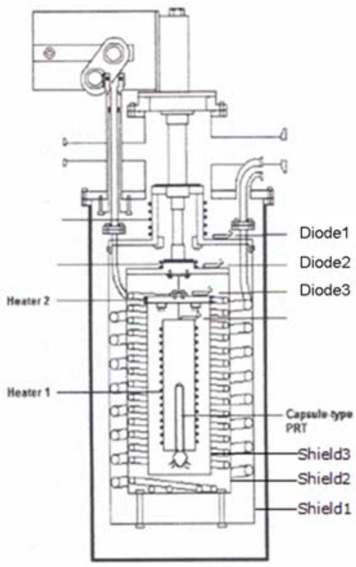

Fig. 1. Schematic diagram of the system for the realization of cryogenic sealed cells.

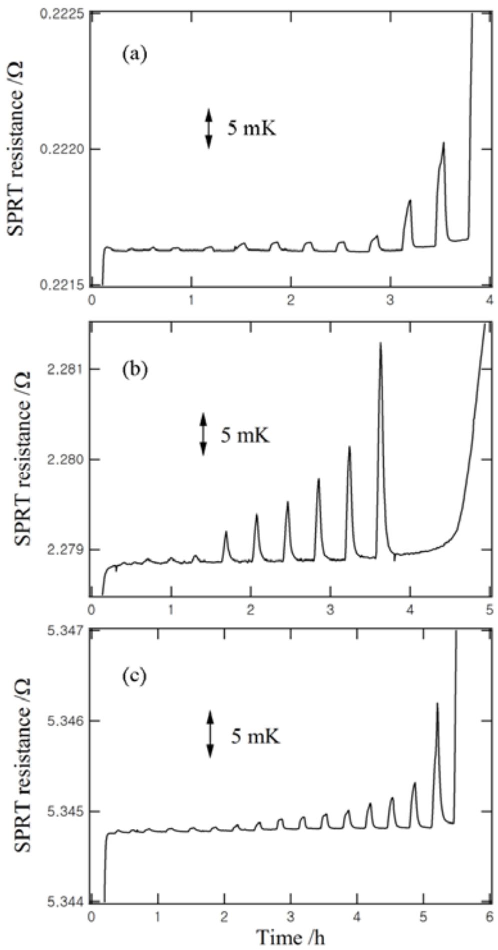

Fig. 2. The result of the adiabatic realization of the triple point of (a) neon (b) oxygen, and (c) argon using heat-pulse method.

Fig. 2 shows the full melting curves of the TPs of Ne, O_2 and Ar, while adiabatic realizations are found using pulse heating. The total heat power throughout the pulse heating was 25 J for Ne, 79 J for O_2, and 140 J for Ar. The uncertainty of the calibration of the capsule-type SPRTs using these cells was within 1 mK. Since operating sealed cells is much easier than operating open cells, they are suitable for the dissemination of the temperature scale through capsule-type SPRTs. Table 1 shows the uncertainty budget of the calibration of capsule-type SPRT by the realization of the O_2 cell.

Table 1. Uncertainty budget of the TP of O_2 by capsule-type SPRT

Uncertainty components	Uncertainties (mK)	Degrees of freedom
Sealed cell uncertainty	0.1	∞
Determination of the plateau value	0.05	∞
Plateau repeatability	0.1	4
Self-heating	0.02	∞
Heat flux	0.15	∞
Standard resistors	0.01	∞
Hydrostatic head correction	0.015	∞
Propagation from TPW uncertainty	0.01	∞
Expanded uncertainty (k = 2)	0.43	

2.3. Sealed cell of the TP of Ar for long-stem SPRTs

Long-stem SPRTs can be calibrated and used for temperature measurements above the TP of Ar or above –200 °C with extrapolation in the range of ~10 K. The TP of the Ar system was designed and used for calibrating long-stem SPRTs [3]. The original design has been modified since then to reduce heat flux from the atmosphere and to ease liquid nitrogen filling. To achieve this, the top flange of the cryostat was lowered to leave more space for the liquid nitrogen above it. The modified cryostat is shown in Fig. 3.

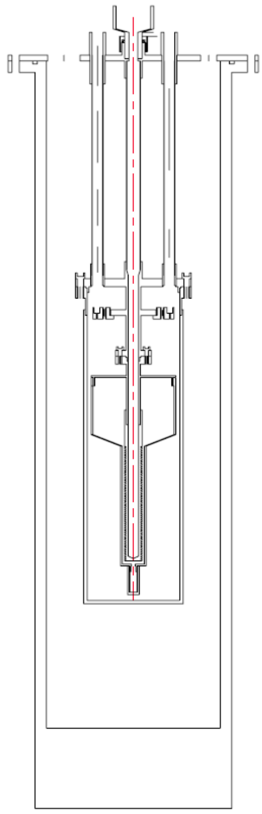

Fig. 3. The modified design of the TP of the Ar system for long-stem SPRTs.

As shown in Table 2, the improved uncertainty of the TP of Ar realization by long-stem SPRT was 0.4 mK (k = 2). The system was used for the ongoing international comparison on the TP of Ar for long-stem SPRTs, APMP.T-K3.2 [4].

Table 2. The uncertainty budget of the TP of Ar by long-stem SPRT

Uncertainty component	Uncertainty (mK)	Degrees of freedom
Determination of the plateau value	0.09	∞
Plateau repeatability	0.1	4
Chemical impurity	0.03	2
Self heating	0.03	∞
Heat flux	0.05	∞
Standard resistor	0.01	∞
Hydrostatic head correction	0.02	∞
Propagation from TPW uncertainty	0.03	∞
Expanded uncertainty (k = 2)	0.38	

2.4. Controllable comparison cryostat

A temperature-controllable cryostat was manufactured for the calibration by comparison of long-stem SPRTs and capsule-type SPRTs. The design for the cryostat was essentially the same as the one the National Research Council of Canada uses [5]. The cryostat was designed so that four capsule-type SPRTs and four long-stem SPRTs can be compared at the same time. We used liquid nitrogen as a coolant, and the base temperature of the cryostat was ~–190 °C when cooled down for about 10 h from room temperature. Fig. 4 shows a photograph of the comparison cryostat. We compared two capsule-type SPRTs calibrated at their sealed cells up to the TP of O_2, and one long-stem SPRT calibrated at the TP cell of Ar for the long-stem SPRT. The measurement showed that the calibration results were consistent within 1 mK near the TP of Ar. The uncertainties of each calibration were in the order of, or less than, 1 mK. The uncertainty of this comparison was larger than 1 mK and was mainly due to the inhomogeneity within the cryostat.

One problem of this comparison cryostat is that its time constant is very large because of the large thermal mass of the copper block. Therefore, it usually takes longer than one hour to stabilize the temperature of the system at a calibration temperature, and for more precise calibration, the required time is even longer. To resolve this problem, a method of dynamic calibration with first-order compensation of the time lag was suggested and successfully tested [6]. As long as the rate of the temperature change of the cryostat is low enough, this method is useful, even when active temperature control of the cryostat is unavailable.

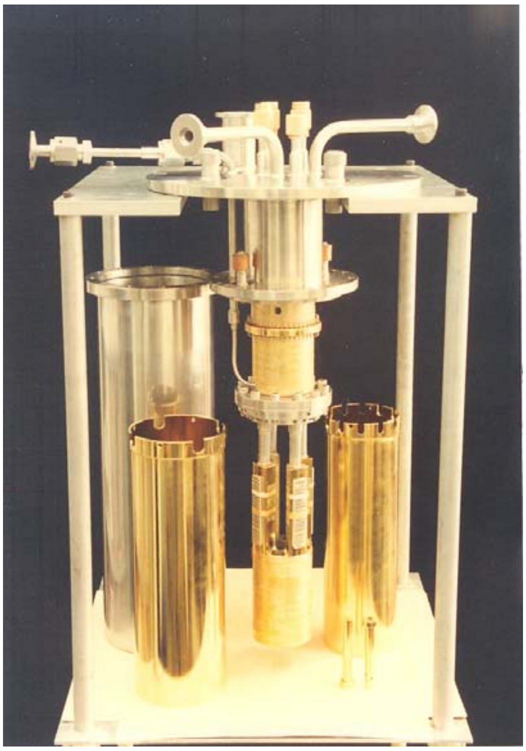

Fig. 4. Photograph of the cryostat for low temperature calibration by comparison.

2.5. Liquid nitrogen block

For customers who need calibration at the boiling point of nitrogen, the comparison cryostat described in Section 2.4 would be superfluous because it gives a calibration result in a given temperature range. We manufactured the boiling point of the nitrogen block calibrator. The stability of the comparison block is 2 mK and it was designed for the comparison calibration of industrial platinum resistance thermometers with a calibrated SPRT. Fig. 5 shows the liquid nitrogen comparison block. Six test thermometers can be compared simultaneously, with an SPRT in the centre.

Fig. 5. Photograph of the liquid nitrogen comparison block.

3. TRACEABILITY SCHEMES

The traceability of the low temperature standards at KRISS will be supported by two international key comparisons, CCT-K2 (completed) and APMP-K3.2 (ongoing). Fig. 6 shows the traceability scheme of our temperature standards below the TP of mercury. Some traceability is completed (solid lines) and some has yet to be done (dashed lines). The traceability starts from the open cells, which is traceable to international temperature scales via CCT-K2. Since we have not submitted the e-H_2 point in CCT-K2, the traceability chain is not complete. The calibration services are done with the sealed cells. For traceability, the sealed cells should be certified by the open cells, and this is still to be done. Long-stem thermometers up to the TP of Ar are calibrated at the sealed Ar cell. The traceability of this Ar cell will be supported by APMP.T-K3.2, which, in turn, will be linked to CCT-K3. The linkage will be provided by NIM (National Institute of Metrology, China) and NIST (National Institute of Science and Technology, USA). The Ar cell has also been compared with the sealed cell, and the result was within 1 mK. Industrial thermometers are calibrated by comparison in the controllable comparison cryostat, either with a reference capsule-type SPRT that is calibrated at the sealed cells or with a long-stem SPRT that is calibrated at the Ar cell for long-stem SPRTs. Furthermore, the liquid nitrogen block is used for comparison at the boiling point of nitrogen. In this case, the reference thermometer is a long-stem SPRT that is calibrated up to the triple point of Ar and extrapolated to the boiling point of liquid nitrogen, or a long-stem SPRT that is compared with the calibrated capsule-type SPRT near the boiling point of nitrogen.

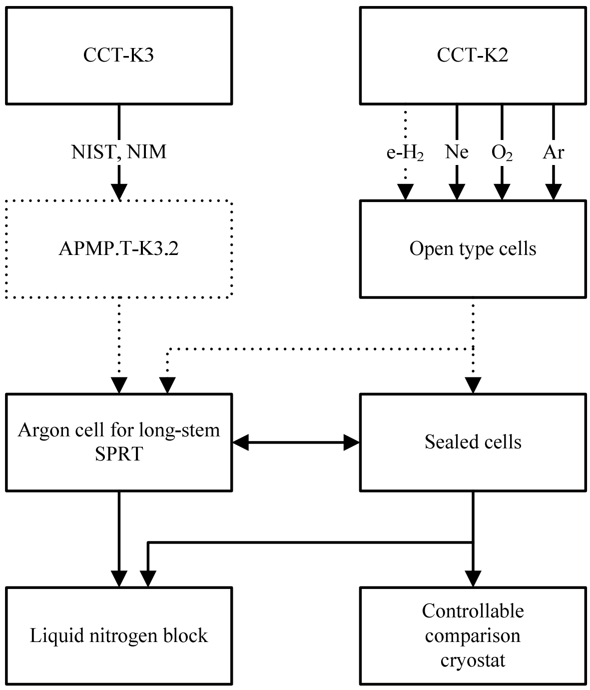

Fig. 6. The traceability scheme of the low temperature standards at KRISS. Solid lines indicate completed links; dotted lines indicate links to be completed in the mid-term.

4. MID-TERM PLANS

Our mid-term (three to five years) goals for low temperature thermometry are to set up calibration facilities for the entire range of SPRTs (13.8033 K and 24.5561 K to ~273.16 K) traceable to international temperature standards. For that, we are working on completing the missing links represented by dotted lines in Fig. 6.

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