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Realization of the Gallium Triple Point at NMIJ/AIST

T. Nakano · O. Tamura · H. Sakurai

Abstract The triple point of gallium has been realized by a calorimetric method using capsule-type standard platinum resistance thermometers (CSPRTs) and a small glass cell containing about 97 mmol (6.8 g) of gallium with a nominal purity of 99.99999%. The melting curve shows a very flat and relatively linear dependence on $1/F$ in the region from $1/F = 1$ to $1/F = 20$ with a narrow width of the melting curve within 0.1 mK. Also, a large gallium triple-point cell was fabricated for the calibration of client-owned CSPRTs. The gallium triple-point cell consists of a PTFE crucible and a PTFE cap with a re-entrant well and a small vent. The PTFE cell contains 780 g of gallium from the same source as used for the small glass cell. The PTFE cell is completely covered by a stainless-steel jacket with a valve to enable evacuation of the cell. The melting curve of the large cell shows a flat plateau that remains within 0.03 mK over 10 days and that is reproducible within 0.05 mK over 8 months. The calibrated value of a CSPRT obtained using the large cell agrees with that obtained using the small glass cell within the uncertainties of the calibrations.

Keywords Gallium · ITS-90 · Triple point

1 Introduction

The triple point of gallium (302.9166 K) has been found to be a highly reproducible fixed point near room temperature [1–11]. Thus, the triple point of gallium is a useful fixed point for realizing the International Temperature Scale of 1990 (ITS-90) [12] and for calibrating standard platinum resistance thermometers (CSPRTs) near room

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temperature [1–11]. The triple point of gallium is usually realized by large cells containing about 1 kg of gallium immersed in a liquid bath maintained near 30°C [1–11]. Although such large cells are convenient for calibrating standard platinum thermometers, strictly speaking, the traditional large cell is far from the definition of the triple point, i.e., the thermal equilibrium state of the solid, liquid, and gas of a pure sample. To check the performance of the calibration using the large cell, it is important to compare the reproducible temperatures realized by large cells with the temperature obtained by an equilibrium state closer to the definition as realized by an adiabatic calorimetric method.

In this study, the triple point of gallium was realized through a calorimetric method using a small glass cell and CSPRTs to closely approximate the ideal equilibrium state of the triple point of gallium, and to investigate the effect of impurities. We also fabricated a large cell for the triple point of gallium, using the same source of material as that used for the small glass cell, for calibrating CSPRTs at the National Metrology Institute of Japan (NMIJ). Moreover, we have checked the performance of the large cell by comparing the calibrated value of a CSPRT obtained using the large cell with that obtained using the small glass cell.

2 Experimental

In this experiment, about 97 mmol (6.8 g) of gallium with a nominal purity of 99.99999% is sealed in a small glass cell about 40 mm in length. The small glass cell for the gallium triple point is prepared in the same way as that for the mercury triple point [13,14]. The triple point of gallium using the small glass cell is realized by the same type of adiabatic calorimeter as was used for the realization of the triple point of mercury [14]. The glass cell is inserted horizontally into a high purity aluminum comparison block. The aluminum comparison block, which is 3 cm in diameter and 6 cm in length, has three holes for the glass cell and two CSPRTs. The comparison block is covered by two temperature-controlled radiation shields and is hung from the upper part of the first radiation shield by nylon strings. The temperature of the first radiation shield is controlled by a PID controller through a DC amplifier using three Type T thermocouples placed in series between the radiation shield and the comparison block. The first radiation shield is also hung by nylon strings from the top of the second radiation shield, whose temperature is controlled using a temperature controller with an industrial grade platinum resistance thermometer. The calorimeter design is described in detail in [14].

The energy for the calorimetry measurement was calculated from the electrical power supplied to the heater (with a resistance of about 300 Ω, and wound around the comparison block) and the duration of heating. The heating current used to realize the triple point was typically about 20 mA.

Figure 1 is a schematic side view of the large gallium triple-point cell used to calibrate CSPRTs at NMIJ. The gallium triple-point cell consists of a PTFE container and a PTFE cap with a re-entrant well and a small vent. The PTFE cell contains 780 g of gallium from the same source as used for the small glass cell. The height between the liquid level and the inner bottom of the thermometer well is 143 mm. The PTFE cell

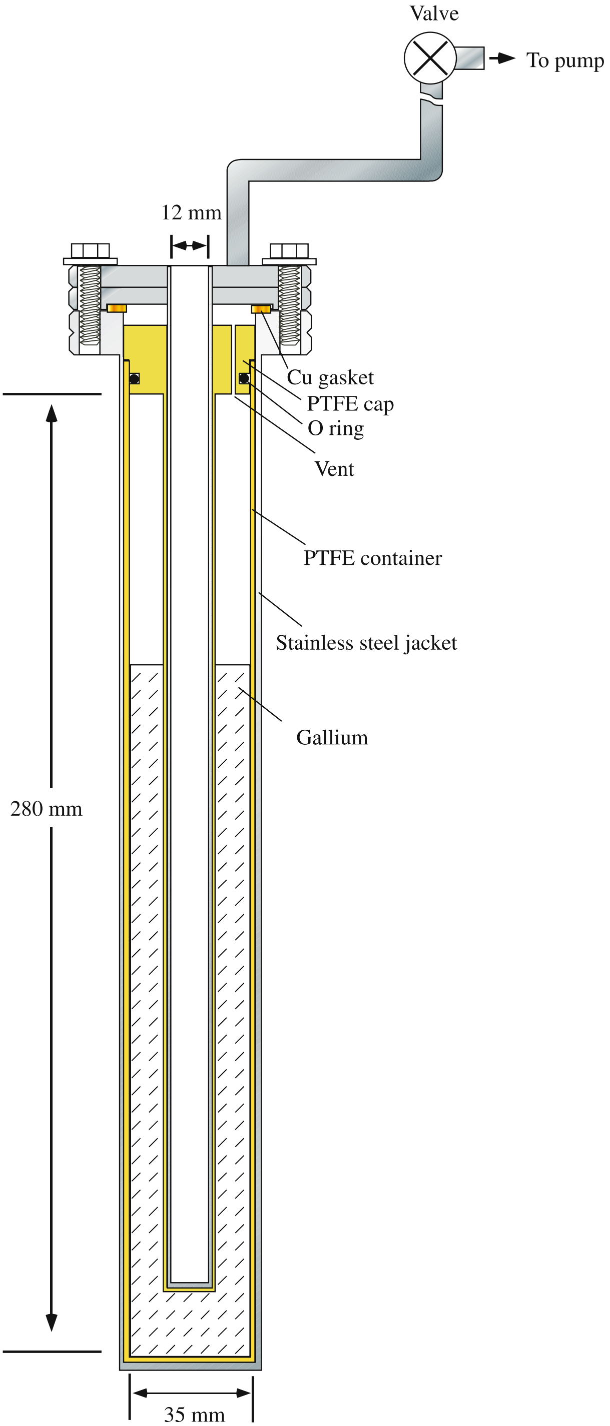

Fig. 1 Schematic side view of the large cell for the realization of the triple point of gallium

is completely covered by a stainless-steel jacket with a valve to evacuate the cell. The gallium triple-point cell was assembled in a nitrogen-filled glove box, and evacuated by an oil-free turbo-molecular pump at 80°C for over 1 week. Then, the gallium was solidified.

The gallium triple point is realized with the large cell by using a water bath that controls the ambient temperature of the cell. The bath temperature is set at a temperature above the gallium triple point during the realization, and the stability of the bath temperature is within 2 mK. To create a liquid–solid interface around the re-entrant well, an immersion heater is placed into the re-entrant well (about 5% of the sample melted) at the beginning of melting in a manner similar to that reported in [8].

The inner diameter of the re-entrant well is 12 mm, into which a sheath assembly of a CSPRT is inserted for calibration. The sheath assembly consists of a stainless-steel tube and a copper block, into which the CSPRT is placed. The detailed structure of the sheath assembly has been reported in [15,16]. For the calibration of CSPRTs, the sheath assembly is set 1 cm above the bottom of the well using an insulating spacer in order to avoid a buoyancy effect on the realized fixed-point temperature as was reported for the realization of the triple point of water using large cells [15–17].

The resistance of the CSPRTs is measured using an automatic direct-current comparator bridge. The excitation current, I, is 1 mA for the measurements of the CSPRTs. The calibration values are given for zero thermometer current, obtained by extrapolating the observation made at I and $\sqrt{2}I$ to correct for self-heating effects.

3 Results and Discussion

Figure 2 shows an example of the melting curves of gallium obtained using the small glass triple-point cell. The figure plots the relationship between the resistance of the thermometer and the inverse of the fraction melted, $1/F$. The melting curve shows a flat and relatively linear dependence on $1/F$ in the region from $1/F = 1$ to $1/F = 20$

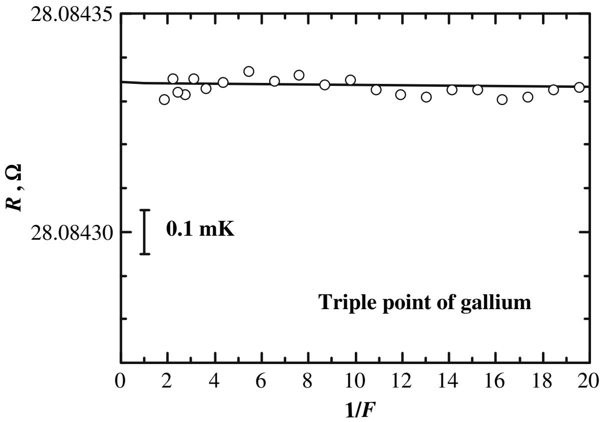

Fig. 2 An example of melting curves at the triple point of gallium obtained by a calorimetric method using a small glass cell and a capsule type standard platinum resistance thermometer

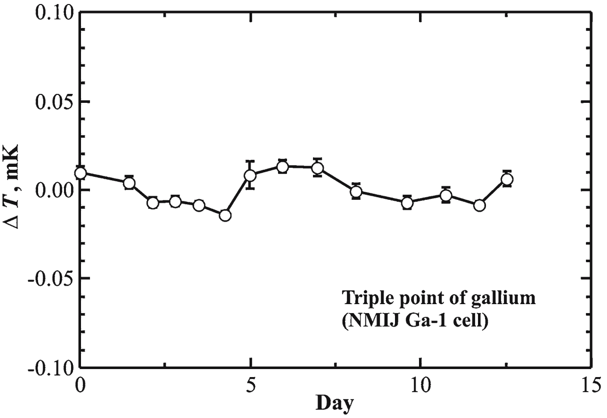

Fig. 3 Stability of the temperatures of the large triple-point-of-gallium cell. The vertical axis plots the deviation of the measured temperatures from the average of the measured temperatures for the large cell

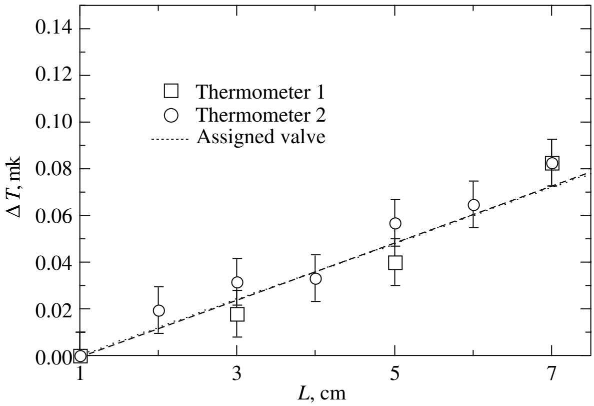

Fig. 4 An example of the immersion characteristics of two capsule-type standard platinum resistance thermometers in the large triple point of gallium cell. The horizontal axis is the distance from the bottom of the well. The vertical axis plots the difference from the temperature at 1 cm from the bottom of the well. The dotted line is the assigned value of the ITS-90 [12]

with a narrow width of the melting curve of <0.1 mK. This behavior is consistent with that expected from samples with higher than 99.99999% purity.

Figure 3 shows an example of the melting curves obtained using the large triple-point cell of gallium, NMIJ Ga-1. A melting plateau is observed extending for 10 days with a narrow width of the melting curve of <0.03 mK. Furthermore, the gallium triple point is reproducible to within 0.05 mK using the large gallium triple-point cell for a period of 8 months.

Figure 4 shows the hydrostatic head effects of the NMIJ Ga-1 cell measured by two CSPRTs. The horizontal axis is the distance from the bottom of the well. The dotted line is the assigned value of the ITS-90 [12]. As seen in Fig. 4, the immersion characteristics of the NMIJ Ga-1 cell agree with the assigned value of the ITS-90 and are consistent with former reports [2,7,9].

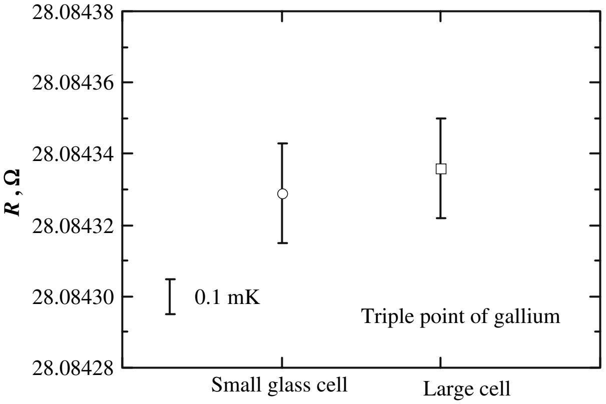

Fig. 5 Comparison of the calibration resistance values for a capsule type standard platinum resistance thermometer obtained using a small glass cell and a large cell. The error bars are the expanded uncertainties for the calibration using the small glass cell and the large cell (Table 1)

Table 1 Uncertainty budgets for the calibration of a capsule-type platinum resistance thermometer at the triple point of gallium obtained using a small glass cell and a large cell

Uncertainty components	Kind of cell	
	Small glass cell	Large cell
Standard uncertainty (mK)		
Realization		
Chemical impurities	0.01	0.01
Hydrostatic head effect	0.002	0.02
Thermal equilibrium	0.04	0.02
Reproducibility	0.04	0.05
Determination of TP value	0.03	0.02
Measurement		
Bridge accuracy	0.01	0.01
Standard resistor	0.02	0.02
Self heating	0.01	0.03
Standard combined uncertainty (mK)	0.07	0.07
Expanded uncertainty ($k = 2$) (mK)	0.14	0.14

Figure 5 shows an example of the calibrated resistance values of a CSPRT obtained using the small glass cell and the large cell. A hydrostatic-head correction is made for the calibrated resistance obtained using the large cell. The error bars represent the expanded standard uncertainty for the calibration performed using both cells, which are listed in Table 1. The two calibrated resistance values agree within the uncertainties of the calibrations. This indicates that our new large cell for the triple point of gallium performs well enough to be used for the calibration of client CSPRTs.

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