Int J Thermophys (2008) 29:31–41
DOI 10.1007/s10765-007-0319-3

NMIJ Constant-Volume Gas Thermometer for Realization of the ITS-90 and Thermodynamic Temperature Measurement

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Published online: 16 January 2008

Abstract The constant-volume gas thermometer (CVGT) of the National Metrology Institute of Japan (NMIJ), AIST with ^{3}He as the working gas is used as an interpolating gas thermometer to realize the International Temperature Scale of 1990 (ITS-90) from 3 K to 24.5561 K and as a relative gas thermometer for thermodynamic temperature measurement calibrated at the triple point (TP) of Ne. The standard uncertainties of the realization and measurement are estimated to be 0.58 mK and 0.86 mK at a maximum in the mentioned temperature range, respectively. The maximum difference between both temperatures is about 1 mK. In the calibration of the CVGT, the TP of equilibrium hydrogen (e-H_2) is corrected for isotopic composition as specified in the Technical Annex for the ITS-90. The ambiguity of the TP of Ne due to the variability in isotopic composition is included in the uncertainty. Although the CVGT was also used in 2004 to realize the ITS-90, it was modified for the present experiment to reduce some measurement uncertainty components and the working gas was replaced with a higher-isotopic-purity gas. The results from 2004 were recalculated by correcting for the isotopic composition of e-H_2 and differ insignificantly from the present results, except for a wider scatter.

Keywords Constant-volume gas thermometer · Helium 3 · ITS-90 · Triple point

1 Introduction

The International Temperature Scale of 1990 (ITS-90) is defined in the temperature range from 3 K to 24.5561 K by interpolation using a constant-volume gas thermometer

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(CVGT) with ^{4}He or ^{3}He as the working gas that is calibrated at three fixed points [1]. A CVGT with this use is called an interpolating constant-volume gas thermometer (ICVGT), and the calibration points are temperatures between 3 K and 5 K determined from the vapor pressure of ^{4}He or ^{3}He (vapor pressure point), the triple point of neon (24.5561 K), and the triple point of equilibrium hydrogen (e-H_2) (13.8033 K). ICVGTs have been constructed and used to realize the ITS-90 at several metrological institutes using ^{4}He as the working gas and some of the realized temperature scales were compared [2–7]. ICVGTs using ^{3}He as the working gas have been constructed in a few cases [8–10].

At the National Metrology Institute of Japan (NMIJ), AIST, a CVGT with ^{3}He as the working gas was constructed for use as an ICVGT. The experimental apparatus, the measurement procedures, the results of some measurement runs, and the realization of the ITS-90 in 2004 from 3 K to 24.5561 K were reported previously [10]. After this realization, the definition of the triple point of e-H_2 as the fixed point of the ITS-90 became more precise with the specification of the isotopic composition of hydrogen to be used and the method to correct the temperature value for samples different from the reference isotopic composition [11]. The scatter of the triple-point temperature of Ne due to the variability in its isotopic composition has also become more evident [12,13]. Meanwhile, at NMIJ, newly manufactured sealed cells have become national reference cells instead of the open cell system used to calibrate the ICVGT in 2004 [14]. Corresponding to these changes, additional corrections and recalculations are made on the temperature scale realized in 2004.

Besides the reanalysis of the previous realization, the CVGT was modified in some structural aspects and used for a new realization of the ITS-90 as reported in this work. To check for possible sample dependence of the scale, the working ^{3}He gas was replaced. The results of the present realization are compared with the previous results recalculated as described above.

CVGTs can be used as relative gas thermometers for thermodynamic temperature measurement requiring calibration at only one fixed point [15–17]. In the CVGT of NMIJ, the temperature profile along the pressure sensing tube is measured to estimate corrections for the dead-space volume and aerostatic pressure head. Therefore, it is possible to conduct a thermodynamic temperature measurement by calibrating the CVGT at only one fixed point instead of the three points of the ITS-90 and by making the explicit corrections that are unnecessary for the interpolating mode. This use of the CVGT as a relative gas thermometer is attempted, and the results are compared with those of the ITS-90 realized simultaneously using the same CVGT as an interpolating gas thermometer.

2 Experimental

The construction of the CVGT and the experimental procedures for use as an interpolating gas thermometer were reported previously [9,10]. Only a brief description is given below.

The bulb containing the working gas at the temperature to be measured is made of oxygen-free high-conductivity copper with a spherical internal volume of about 11 and is enclosed by a radiation shield kept at a temperature slightly below that of the bulb. The pressure in the bulb is measured using a pressure gauge at room temperature through a pressure sensing tube made of stainless steel with an inner diameter of about 2.2 mm and a length of about 1.4 m between the bulb and the room temperature anchor point. The temperature profile along the sensing tube is measured using eight thin-film rhodium–iron resistance thermometers. The temperature profiles between the thermometers are calculated from the thermal conductivity of stainless steel. The bulb is filled with the working ^{3}He gas with a pressure of about 34 kPa at 24 K, that is, a density of about $170\,\text{mol}\cdot\text{m}^{-3}$.

The pressure gauge is a diaphragm-type differential pressure gauge placed in an enclosure whose temperature is regulated. The reference pressure side of the gauge is evacuated to operate the gauge as an absolute pressure gauge. The pressure gauge is calibrated against the pressure generated by a pressure balance, as described in our previous report [10]. Corrections are made to compensate for the effects of the aerostatic pressure head, the thermomolecular pressure difference, and the dead-space volume due to the pressure sensing tube and the pressure gauge based on the measured temperature profile along the pressure sensing tube. The correction for the thermomolecular pressure difference is made using an equation given in the literature [18].

Rhodium–iron resistance thermometers (RIRTs; Type 5187W, H. Tinsley & Co., Ltd.) are inserted into holes on the bulb wall. The resistances of the RIRTs are measured using an automatic direct current resistance ratio bridge and a reference standard resistor of 10 Ω. Excitation currents of 0.5 mA and 0.7071 mA are used in each measurement, and the resistances are extrapolated to the values at zero power to correct for self-heating effects.

The modifications to the CVGT for the present experiment that cause the apparatus to differ from the one used in 2004 are as follows. A thermal anchor was added to reduce heat flow through the pressure sensing tube to the bulb. The thermal anchor connects part of the sensing tube about 6 cm from the bulb with the radiation shield surrounding the bulb. The temperature profile along the sensing tube is kept monotonic by adjusting the strength of the thermal link. The dead-space volume at room temperature was reduced by about 4.5 ml to minimize the dead-space volume correction. The temperature control stability of the pressure gauge was improved to reduce the uncertainty due to the temperature dependence of the gauge. The working gas ^{3}He was replaced to check for possible sample dependence of the realized temperature scale. The isotopic purities are 99.9995% and 99.9999% for the previous and present experiments, respectively.

As calibration points for the ICVGT, a ^{4}He vapor pressure point near 3 K and the triple points of e-H_2 and Ne are used. The RIRTs are calibrated directly by realizing these fixed points, and the CVGT is calibrated against the RIRTs. The realization of the ^{4}He vapor pressure point and the estimated calibration uncertainty of the RIRTs were reported previously [19]. The realization of the triple points of e-H_2 and Ne and the estimation of the calibration uncertainties of the RIRTs at these points are similar to those reported elsewhere [14]. The triple-point temperature of e-H_2 is corrected using the analyzed isotopic composition of the sample hydrogen, as specified in the Technical Annex for the ITS-90 [11]. The uncertainty of the triple point of Ne as estimated includes the uncertainty component due to the ambiguity of the isotopic composition of Ne that is yet to be specified explicitly in the definition of the fixed point [13].

The previous realization of the ICVGT scale in 2004 was reported on the basis of the triple points of e-H_2 and Ne realized using an open cell system without applying isotopic corrections [10]. In this work, these fixed points are realized using newly manufactured national reference sealed cells [14]. The results of the previous realization of the ICVGT scale have been maintained on RIRTs and are now recalculated using the triple-point temperatures of the new sealed cells and by correcting for the isotopic composition of hydrogen as specified in the Technical Annex for the ITS-90.

The CVGT is also used simultaneously as a relative gas thermometer for thermodynamic temperature measurement when calibrated at only one fixed point, the triple point of Ne. The temperature assigned in the ITS-90 is used as the triple-point temperature of Ne for the calibration. Corrections are made for aerostatic pressure head, thermomolecular pressure difference, dead-space volume, deviation from the ideal gas, the thermal expansion of the bulb, and the pressure dilatation of the bulb. The corrections for the first three effects are made in the same way described above for the ICVGT. The correction for the deviation from the ideal-gas law is made using the virial coefficients given in the text of the ITS-90 [1]. The correction for the bulb volume change due to thermal expansion is made using thermal expansion data given in the literature [20,21]. The correction for the bulb volume change due to pressure dilatation is made using elastic constants given in the literature [22]. The effects of the adsorption of the working gas and impurities in the working gas are included in the uncertainties, based on discussions in the literature [15,23].

The measurements using the CVGT in the entire temperature range from 3 K to 24.5561 K were repeated three times in the present experiment. In two of them, the measured temperature changed downwards; in the other one, upwards. In each of the three runs, the CVGT was calibrated at the fixed-point temperature(s) maintained by the RIRT.

In the present measurement of the CVGT, an RIRT (serial number: A67) calibrated at the National Physical Laboratory (NPL) of the United Kingdom in 1990 was also inserted into one of the holes on the bulb wall. The calibration by NPL was reported to be based on the ITS-90 with an uncertainty not greater than 1 mK at the 95% confidence level and an excitation current of 0.3 mA. In this work, the self-heating effect is measured at each measured temperature and the measured resistance is converted to that at an excitation current of 0.3 mA and then the temperature reported by NPL is compared with that realized at NMIJ.

3 Results and Discussion

3.1 Interpolating Gas Thermometer

The estimated uncertainties are summarized in Table 1 for the present realization of the ITS-90 from 3 K to 24.5561 K using the ICVGT. The uncertainty component due to the reference standard pressure generated by the pressure balance is dominant in the uncertainty related to the calibration of the pressure gauge and is shown in Table 1.

Table 1 Uncertainties of realization of the ITS-90 from 3 K to 24.5561 K. The two columns for standard uncertainty represent the minimum and maximum of each uncertainty component in the temperature range. The bottom line represents the minimum and maximum of the combined uncertainty in the temperature range

Uncertainty component	Standard uncertainty (mK)	
	Min	Max
Pressure gauge		
Reference standard pressure	0.14	0.25
Temperature dependence	0.00	0.02
Reproducibility and hysteresis	0.17	0.17
Fluctuation during measurement	0.07	0.17
Corrections in pressure		
Thermomolecular pressure difference	0.00	0.04
Aerostatic pressure head	0.01	0.06
Dead-space volume	0.23	0.24
Resistance thermometer measurement		
Reference standard resistor	0.00	0.01
Resistance ratio bridge	0.01	0.07
Self-heating effect and fluctuation during measurement	0.01	0.05
Temperature gradient	0.03	0.03
Propagation of uncertainties of calibrations at three fixed points	0.27	0.43
Combined uncertainty	0.43	0.58

The uncertainty due to the instability of the pressure gauge is estimated as a long-term component and a short-term one, which correspond to "reproducibility and hysteresis" and "fluctuation during measurement" in Table 1, respectively. To check for uncertainties of the bridge, the resistances of the RIRTs are also measured using a different resistance ratio bridge at several temperatures between 3 K and 25 K. The accuracies of the two bridges should be similar, according to the specifications given by the manufacturers. The difference between the resistances obtained using the two bridges is larger than that expected from the bridge accuracies given by the manufacturers. Therefore, the uncertainty component due to the resistance ratio bridge is estimated from this difference.

In this report, an RIRT (serial number: B271) is used to calibrate the CVGT. The standard uncertainties of the calibrations of the RIRT at the ^{4}He vapor pressure point and the triple points of e-H_2 and Ne are estimated in the same way as published elsewhere [14,19]; they are 0.082 mK, 0.055 mK, and 0.182 mK (standard uncertainty, $k = 1$), respectively. The uncertainties of the calibrations of the ICVGT at these fixed points are obtained by combining these uncertainties and the uncertainties of the comparisons between the RIRT and the CVGT at the fixed-point temperatures; they are 0.34 mK, 0.35 mK, and 0.42 mK (standard uncertainty, $k = 1$), respectively. The calibration uncertainty for the ICVGT is much larger than that for the RIRT because of the uncertainty components related to the pressure measurements. The uncertainties of the

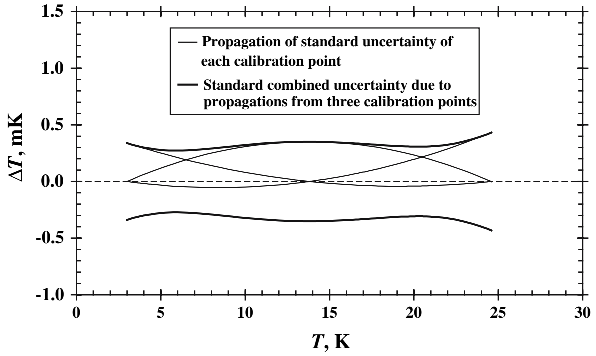

Fig. 1 Propagation of uncertainties of calibration points of ICVGT for realizing the ITS-90. The calibration points are a ^{4}He vapor pressure point near 3 K and the triple points of e-H_2 and Ne. Thick curves represent the combined standard uncertainty due to the propagation from the uncertainties of calibrations at these fixed points. Each of the three thin curves represents how the temperature shift equal to the standard uncertainty of calibration at each fixed point propagates to the interpolated temperature

calibrations of the ICVGT at the three fixed points propagate to all the temperatures obtained from the interpolation, as illustrated in Fig. 1. Each of the thin curves in Fig. 1 represents how a temperature shift equal to the standard uncertainty of calibration at each fixed point propagates to the interpolated temperatures. The thick curves in Fig. 1 represent the combined standard uncertainty due to the propagation of the calibration uncertainties at the three fixed points.

Figure 2 shows the uncertainties of the realization of the ITS-90 using the ICVGT. The curves in Fig. 2 show the combined standard uncertainties of the present realization of the ITS-90 that correspond to the combined uncertainty in Table 1. The $T_{90\mathrm{FIT}}$ denotes the temperature obtained from a 10th-order polynomial of the RIRT resistance fitted to the temperature $T_{90\mathrm{MEAS}}$ measured using the ICVGT in three runs of the present realization. The open symbols in Fig. 2 show the deviation of the measured temperatures $T_{90\mathrm{MEAS}}$ from the fitted temperature $T_{90\mathrm{FIT}}$. The closed symbols in Fig. 2 represent the difference $T_{90\mathrm{in}2004} - T_{90\mathrm{FIT}}$, where $T_{90\mathrm{in}2004}$ denotes the temperature obtained from the realization of the ITS-90 using the ICVGT in 2004 and recalculated as described above. The temperature of the present realization of the ITS-90 agrees with that of the previous realization in 2004 within the standard uncertainty of the present realization. There is no significant systematic difference observed between the temperatures of the two realizations in spite of the modifications in the CVGT described above. Therefore, the reproducibility of the realization is confirmed. However, the temperatures of the previous realization have a greater scatter than those of the present realization. The modifications to the CVGT are credited with the improved performance.

The open symbols in Fig. 3 represent the difference $T_{90\mathrm{NPL}} - T_{90\mathrm{FIT}}$, where $T_{90\mathrm{NPL}}$ denotes the temperature obtained from the resistance-temperature equation assigned by NPL to the RIRT (serial number: A67) in 1990 based on the ITS-90 [24]. The temperatures obtained from the two equations show some systematic difference with a maximum of about 0.7 mK. This difference is within the maximum uncertainty of

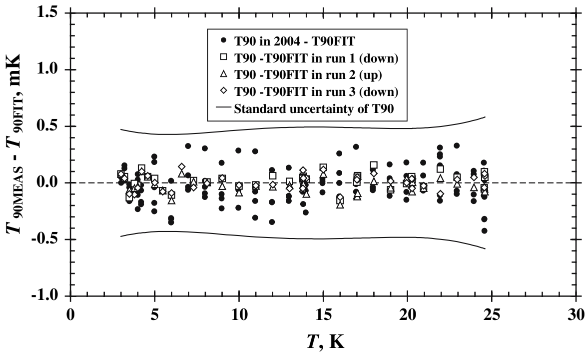

Fig. 2 Uncertainties of realization of the ITS-90 using ICVGT. Curves represent the combined standard uncertainty of the realization of the ITS-90. A 10th-order polynomial of the resistance of an RIRT, $T_{90\mathrm{FIT}} = f(R)$, is fitted to the temperature $T_{90\mathrm{MEAS}}$ obtained from the ICVGT in three runs. Open symbols represent the fit residual $T_{90\mathrm{MEAS}} - T_{90\mathrm{FI}}$. In the legend, "down" means the run with a downward temperature change and "up" means that with an upward temperature change. Closed symbols represent the deviation of the recalculated realization of the ITS-90 in 2004 from $T_{90\mathrm{FIT}}$

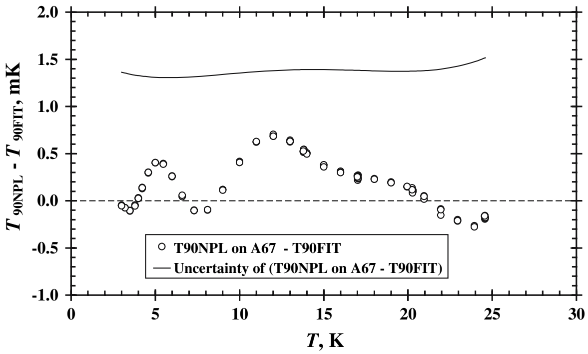

Fig. 3 Comparison with calibration by NPL. Open symbols represent the difference $T_{90\mathrm{NPL}} - T_{90\mathrm{FIT}}$, where $T_{90\mathrm{NPL}}$ denotes the temperature obtained from an RIRT (serial number: A67) calibrated by NPL in 1990 with an uncertainty not greater than 1 mK at the 95% confidence level based on the ITS-90 [24]. Curve represents the square root of the sum of squares of 1 mK and the uncertainty of $T_{90\mathrm{FIT}}$ at the 95% confidence level. The uncertainty of $T_{90\mathrm{NPL}} - T_{90\mathrm{FIT}}$ at the 95% confidence level is not greater than that in this curve

1 mK of the calibration at the 95% confidence level reported by NPL [24]. The calibration by NPL in 1990 is thought not to have taken into account the isotopic composition of the sample hydrogen for the triple-point realization. To realize the triple point of e-H_2 at NMIJ, the NMIJ H-5 cell is used [14]. The triple-point temperature difference between NMIJ H-5 and an e-H_2 triple-point cell made by NPL, H_2-1, has been reported [25]. From this difference, the triple point of the H_2-1 cell is estimated to be 0.31 mK lower than the temperature obtained after correcting the triple point of NMIJ H-5 cell according to the Technical Annex for the ITS-90. This difference is expected to contribute to the $T_{90\mathrm{NPL}} - T_{90\mathrm{FIT}}$ difference of about 0.7 mK at approximately 13 K.

Table 2 Uncertainties of thermodynamic temperature measurement from 3 K to 24.5561 K by calibrating the CVGT at the triple point of Ne. The two columns for standard uncertainty represent the minimum and maximum of each uncertainty component in the temperature range. The bottom line represents the minimum and maximum of the combined uncertainty in the temperature range

Uncertainty component	Standard uncertainty (mK)	
	Min	Max
Pressure gauge		
Reference standard pressure	0.14	0.25
Temperature dependence	0.00	0.02
Reproducibility and hysteresis	0.17	0.17
Fluctuation during measurement	0.07	0.17
Corrections in pressure		
Thermomolecular pressure difference	0.00	0.04
Aerostatic pressure head	0.01	0.01
Dead-space volume	0.23	0.73
Resistance thermometer measurement		
Reference standard resistor	0.00	0.01
Resistance ratio bridge	0.01	0.07
Self-heating effect and fluctuation during measurement	0.01	0.05
Adsorption	0.06	0.06
Impurity	0.07	0.07
Temperature gradient	0.03	0.03
Propagation of uncertainty of calibration at triple point of Ne	0.08	0.66
Combined uncertainly	0.47	0.86

3.2 Relative Gas Thermometer

The estimated uncertainties are summarized in Table 2 for the thermodynamic temperature measurement from 3 K to 24.5561 K after calibrating the CVGT at the triple point of Ne. The standard uncertainty of the thermodynamic temperature of the triple point of Ne defined by the ITS-90 is estimated to be 0.5 mK in the Supplementary Information for the ITS-90 [26]. This estimation is consistent with the thermodynamic temperature data evaluated recently by Working Group 4 of the Consultative Committee for Thermometry [27], which also includes recent measurement results [28,29]. Therefore, in this work, 0.5 mK is adopted as the standard uncertainty of the thermodynamic temperature of the triple point of Ne. For the calculation of uncertainty propagation, the standard uncertainty of the calibration of the CVGT at the triple point of Ne is estimated to be 0.78 mK by combining the uncertainty of 0.41 mK obtained above for the realization of the ITS-90 with the uncertainty of the thermodynamic temperature, 0.5 mK.

The thick curves in Fig. 4 show the combined standard uncertainties of the temperature measurement using the relative CVGT and correspond to the combined uncertainty in Table 2. The thin curves in Fig. 4 show the combined standard uncertainties of the

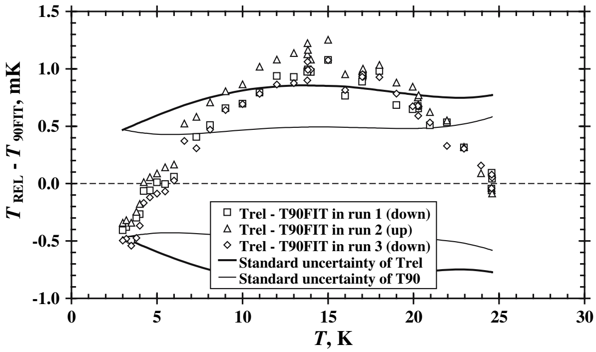

Fig. 4 Difference between relative gas thermometer and the ITS-90. Open symbols represent the difference $T_{REL} - T_{90FIT}$, where T_{REL} denotes the thermodynamic temperature measured by calibrating the CVGT at the triple point of Ne. In the legend, "down" means the run with a downward temperature change and "up" means that with an upward temperature change. The thick and thin curves represent the combined standard uncertainties of T_{REL} and the realization of the ITS-90, respectively

present realization of the ITS-90. The open symbols in Fig. 4 show the difference $T_{REL} - T_{90FIT}$, where T_{REL} denotes the thermodynamic temperature obtained from the relative gas thermometer. The data of $T_{REL} - T_{90FIT}$ show a curve with an upward convex shape with a maximum of about 1 mK, which is within the combined expanded uncertainty (with a coverage factor of 2) of the present measurement of T_{REL}. However, this curve resembles qualitatively the differences of the thermodynamic temperature from the ITS-90 that were reported recently using a dielectric-constant gas thermometer and an acoustic gas thermometer [28,29].

4 Conclusion

The CVGT with ^{3}He as the working gas is used simultaneously as both interpolating and relative gas thermometers in the temperature range from 3 K to 24.5561 K. The standard uncertainties of the realization of the ITS-90 using the interpolating gas thermometer are estimated to be 0.58 mK (maximum) in the temperature range. The standard uncertainties of the thermodynamic temperature measurement using the relative gas thermometer are estimated to be 0.86 mK (maximum) in the temperature range, including the propagation of the uncertainty at the triple point of Ne. The difference from the ITS-90 shows a curve with an upward convex shape with a maximum of about 1 mK in the temperature range, although it is within the combined expanded uncertainty (with a coverage factor of 2) of the thermodynamic temperature measurement.

The realization of the ITS-90 in 2004 recalculated by adopting the same triple-point temperatures used for the present realization has no significant deviation from the present one, except for a wider scattering of the temperatures than in the present case. The difference between the realization of the ITS-90 at NMIJ and the RIRT calibrated by NPL on the ITS-90 in 1990 is less than 0.7 mK in the temperature range, which is within the possible combined uncertainties of the difference at the 95% confidence level.

Acknowledgments The authors thank Dr. T. Shimazaki and Dr. H. Nakagawa for valuable discussion.

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