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Isotopic Correction of Water-Triple-Point Cells at NMIJ

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Abstract The twenty-one participating laboratories in the international key comparison of water-triple-point cells (CCT-K7) can be classified into three groups: two laboratories that corrected the effect of the isotopic composition of water, four laboratories that had information on the isotopic composition but did not correct the effect, and the remaining laboratories that had no information. There were significant differences in the realized national standard for the triple point of water (TPW) between those laboratories that applied the isotopic correction and those that did not. The isotopic correction is now considered essential for the triple point of water. Since the National Metrology Institute of Japan (NMIJ) did not apply the isotopic correction and estimated large uncertainties at the time of the CCT-K7 comparison, we subsequently developed new cells for the TPW to improve the reliability and to reduce the uncertainty of the realization as a national reference. The isotopic compositions of seven cells were analyzed, and a chemical impurity analysis of one cell was performed. The good consistency among seven cells was shown in the results obtained when the isotopic correction was applied to the realized temperatures measured experimentally. The expanded uncertainty of the new national reference of NMIJ is estimated to be 49 μK ($k = 2$), and as a result of this improvement, the expanded uncertainty for calibrating a water-triple-point cell is 80 μK. The previous reference of NMIJ, reported in CCT-K7 to have an expanded uncertainty of 302 μK, is 42 μK lower than the new one. The new reference value is within the uncertainty of the previous national reference, and the new uncertainty is completely covered by the previous uncertainty. Furthermore, the new reference of NMIJ shows good agreement with the national references of the six

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laboratories able to apply isotopic corrects to their results for CCT-K7. These facts confirm the validity and the linkage to the CCT-K7 of both the previous and the new national references of NMIJ.

Keywords Isotope · National standard · Temperature · Triple point of water · Uncertainty

1 Introduction

The international key comparison of water-triple-point cells, CCT-K7 (Consultative Committee for Thermometry Key Comparison 7), was conducted with the International Bureau of Weights and Measures (BIPM) as the pilot laboratory and twenty-one participating laboratories [1]. Figure 1 shows a graph that the authors reproduced from Table 21 of the final report, along with additional information. The laboratories can be classified into three groups: those that applied the isotopic correction based on measured ratios of D/^{1}H (where symbol D designates ^{2}H) and ^{18}O/^{16}O, those that had information on the isotopic composition but did not apply the correction, and those that had no information. In Fig. 1, the values of the second group were corrected using their information regarding the isotopic composition. There is a distinct temperature difference of 87 μK between the corrected values (the first and the second groups) and the uncorrected values (the third group).

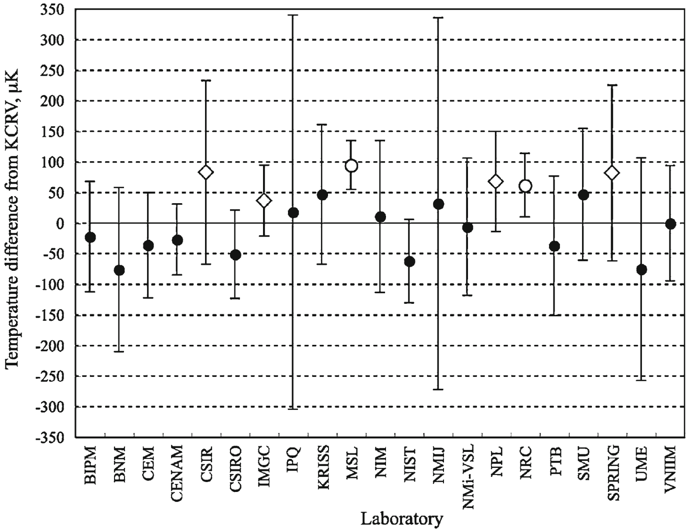

Fig. 1 Results of the international key comparison of water-triple-point cells (CCT-K7). The values are reproduced by combining Tables 21 and 22 of the final report [1], along with the classification of the status of the isotopic correction. ○: laboratories that applied the isotopic correction. ◇: laboratories that did not apply the isotopic correction although they reported the isotopic compositions. The values have been corrected by the authors using this information. ● : laboratories that did not apply the isotopic correction and reported no information regarding isotopic compositions

When the CCT-K7 was performed, the isotopic composition of water for the TPW was defined as the same composition as Standard Mean Ocean Water (SMOW) in the "Supplementary Information for the International Temperature Scale of 1990" [2]. However, the majority of laboratories participating in the CCT-K7 did not apply the isotopic correction, and only six laboratories reported the isotopic composition. The simple mean of the participating laboratories was adopted as the key comparison reference value (KCRV) for the CCT-K7 as a result of a vote among the participants. The isotopic correction, however, is now considered essential for the TPW, since the result of the CCT-K7 showed that the temperatures reported by many participants without the isotopic correction were distinctly lower than those reported with the isotopic correction. Additionally, the description of the TPW in regard to the definition of the kelvin in the International System of Units (SI) was revised to strictly adopt the isotopic composition of V-SMOW (Vienna Standard Mean Ocean Water) at the CIPM (International Committee for Weights and Measures) meeting in 2005 [3].

The National Metrology Institute of Japan (NMIJ), one of the participants in the CCT-K7, neither applied the isotopic correction nor analyzed the water of the national reference cells for chemical impurities and residual gases, at that time. Although we understood that these effects were not negligible, we had no reliable information regarding the isotopic compositions and impurities. Consequently, we estimated the uncertainty of the national reference using the literature values [4], which resulted in a relatively large uncertainty. The influence of isotopic variations and chemical impurities was estimated to be 100 μK, and the combined standard uncertainty 151 μK.

After the CCT-K7, we developed new national reference cells for the TPW to improve the reliability of the TPW realization temperature. We conducted analyses of the isotopic composition and chemical impurities, and comparison measurements of the realization temperatures were made among our new cells.

2 Preparation of TPW Cells

The seven cells were prepared as identified in Table 1. The glass of cell No. 4 is fused silica, and that of the others borosilicate glass. Some cells have a pressure tube, whose inner diameter and length are 5 mm and 25 mm, respectively. This is used as a small McLeod gauge to evaluate the internal gas pressure [5].

The isotopic composition of the water in cell No. 4 was adjusted by the manufacturer to be close to that of V-SMOW. Cell Nos. 3, 5, 6, and 7 were produced by collaboration between Toa Keiki Mfg. Co., Ltd. (Tokyo, Japan) and NMIJ. Cell Nos. 6 and 7 were made from distilled water infused with a small amount of heavy water. In order to sample the water in the cells, each cell (Nos. 3–7) included a small ampoule when they were manufactured. After filling and sealing, the ampoule was filled with the encapsulated water and removed without breaking the seal of the cell. The isotopic compositions of the water in the ampoules were analyzed. Cell Nos. 1 and 2 had no ampoule, and they were broken after the thermal measurements to analyze the water.

Table 1 Specification of water-triple-point cells

Cell No.	S/N	Manufacturer	Year of fabrication	Inner diameter of well (mm)	Cell diameter (mm)	Depth of well below water Surface (mm)	Pressure tube
1	15146	TOA KEIKI MGF.	2003	10	65	260	Without
2	16161	TOA KEIKI MGF.	2004	12	65	255	Without
3	17165	TOA KEIKI MGF. & NMIJ	2005	10	65	255	With
4	Q1008	Hart Scientific	2005	12	60	265	Without
5	T2006-1	TOA KEIKI MGF. & NMIJ	2006	10	65	255	With
6	T2006-2	TOA KEIKI MGF. & NMIJ	2006	12	60	260	With
7	T2006-3	TOA KEIKI MGF. & NMIJ	2006	12	60	255	With

A heat-pipe cooling instrument and liquid nitrogen were used to prepare the ice mantle in the cell. The cells were kept in a temperature-controlled water bath. To stabilize the ice mantle, the temperature measurements were performed one week after its preparation.

3 Qualification of TPW Cells

3.1 Isotopic Composition

The isotopic analysis of the water in the cells was carried out by SHOKO Co., Ltd (Saitama, Japan). To confirm the validity of the analysis and to estimate the uncertainty, water samples from the cells and additional samples were analyzed by the following three laboratories: Forestry and Forest Products Research Institute (FFPRI, Tsukuba, Japan), Toray Research Center, Inc. (Otsu, Japan), and the University of Utah (Salt Lake City, USA). The FFPRI was one of the participants in the “Third interlaboratory comparison exercise δD and δ^{18}O analysis water samples (WICO2002-Part I)” organized by the International Atomic Energy Agency (IAEA) [6].

Variations in isotope amount-of-substance ratios are conventionally reported as deviations from V-SMOW. For example:

$$\delta^{18}\mathrm{O} = \left[\left({}^{18}\mathrm{O}/{}^{16}\mathrm{O}\right)_{\text{sample}} - \left({}^{18}\mathrm{O}/{}^{16}\mathrm{O}\right)_{\text{V-SMOW}}\right] / \left({}^{18}\mathrm{O}/{}^{16}\mathrm{O}\right)_{\text{V-SMOW}},$$

and similarly for δD and δ^{17}O. The results of the analysis of the cells are presented in the parts-per-thousand range expressed in per mil (per thousand, $^{0}/_{00}$) in Table 2. The additional water samples X, Y, and Z were also analyzed. The water of the samples X and Y consisted of commercially available distilled water, and that of sample Z was

Table 2 Results of isotopic analysis

Cell No.	S/N	Labaratory for analysis	Results ‰			Estimation of temperature difference from V-SMOW (μK)
			δD	δ^{17}O	δ^{18}O	
1	15146	FFPRI[a]	−50.2	−4.22	−7.97	−36.9
		SHOKO Co., Ltd.	−48.7	−4.17	−7.88	−35.9
2	16161	SHOKO Co., Ltd.	−47.6	−3.93	−7.43	−34.9
3	17165	SHOKO Co., Ltd.	−70.2	−5.15	−9.73	−50.6
4	Q1008	SHOKO Co., Ltd.	1.7	−0.11	−0.20	1.0
		University of UTAH	2.0	−0.16	−0.30	1.1
5	T2006-1	SHOKO Co., Ltd.	−69.4	−5.21	−9.84	−50.2
6	T2006-2	SHOKO Co., Ltd.	36.3	−5.82	−11.00	15.4
7	T2006-3	SHOKO Co., Ltd.	8.8	−5.45	−10.30	−1.4
Water sample X		SHOKO Co., Ltd.	−58.1	−4.74	−8.96	−42.5
		Toray Research Center	−60	−4.87	−9.2	−43.9
Water sample Y		SHOKO Co.,Ltd	−51.0	−4.29	−8.11	−37.5
		Toray Research Center	−50	−4.34	−8.2	−36.9
Water sample Z		FFPRI[a]	58.4	−4.47	−8.45	31.0
		SHOKO Co., Ltd.	61.5	−4.49	−8.48	32.9

[a] Forestry and Forest Products Research Institute

distilled water infused with a small amount of heavy water. The value for δ^{17}O was not measured, but estimated using the following equation [7]:

$$1 + \delta^{17}\mathrm{O} = (1 + \delta^{18}\mathrm{O})^{0.528} \tag{1}$$

The temperature differences from V-SMOW were estimated using [8,9]

$$\Delta T = T_{\mathrm{meas}} - T_{\mathrm{V\text{-}SMOW}} = A_{\mathrm{D}}\delta\,\mathrm{D} + A_{17\mathrm{O}}\delta^{17}\mathrm{O} + A_{18\mathrm{O}}\delta^{18\mathrm{O}} \tag{2}$$

Depression constants derived from Kiyosawa [8] were used as follows: $A_{\mathrm{D}} = 628\,\mu\mathrm{K}$, $A_{18\mathrm{O}} = 641\,\mu\mathrm{K}$, and $A_{17O} = 57\,\mu\mathrm{K}$.

The uncertainty of the isotopic correction consists of four components. The first one is the repeatability of the measurements of a water sample, and this is evaluated by the standard deviation of repeated measurements for the same sample. The standard uncertainty in the measured values of δD and δ^{18}O are 0.5 ‰ and 0.04 ‰, respectively, and these were transformed to temperature equivalents of 0.31 μK and 0.03 μK, respectively.

The second component is the reproducibility among laboratories. In the report of the third interlaboratory comparison exercise (WICO2002-Part I), it is stated that the standard uncertainties of the values of the participating laboratories are 1.7 ‰ for δD and 0.15 ‰ for δ^{18}O. In the present study, some samples were analyzed by two different laboratories and the maximum differences are 3.1 ‰ for δD and 0.24 ‰ for δ^{18}O. If it is assumed that the probability distributions of the measurements are rectangular distributions of width given by these maximum differences, the uncertainties are less than those in the report. Considering these results, the standard uncertainties of the reproducibility among laboratories of δD and δ^{18}O are estimated to be 1.1 μK and 0.1 μK, respectively, calculated from the values of the report.

The third component of the isotopic correction is the uncertainty of the measurements due to systematic error. In the WICO2002-Part I, the standard uncertainties of the reference values determined by IAEA are 0.6 ‰ for δD and 0.04 ‰ for δ^{18}O. The standard uncertainties of the systematic components of δD and δ^{18}O are estimated to be 0.4 μK and 0.03 μK, respectively, after transforming to temperature.

The fourth component is the uncertainty of Eq. 2, which is estimated from the uncertainties of the depression constants. The uncertainties of A_{D} and $A_{18\mathrm{O}}$ are 20 μK and 50 μK, respectively [9], and influence the temperature by 1.4 μK and 0. 5 μK, respectively, within the range of water compositions measured. Since the value of $A_{17\mathrm{O}}$ is quite small relative to the other constants, the uncertainty of $A_{17\mathrm{O}}$ is negligible.

From the above facts, the combined standard uncertainty of the isotopic correction is estimated to be 1.9 μK.

3.2 Chemical Impurities

To estimate the effect of the chemical impurities dissolved in the water of the cells, quantitative analyses for B and Na and qualitative analyses for other elements were undertaken using the actual water from a cell with an inductively coupled plasma mass spectrometer (ICPMS). Quantitative analysis for Si was performed with an atomic absorption spectrometer. After the temperature comparison measurements (described later), the water in cell No. 1 was collected just after the cell was broken, and then analyzed by Toray Research Center.

Since it is expected that Si, B, and Na are the dominant impurities dissolved from borosilicate glass, these elements received quantitative analysis, whereas the other elements received a qualitative analysis. The results of the analyses are shown as the concentrations of impurities in Table 3.

The depression of the triple point of water by an impurity is estimated thermodynamically as follows:

$$\Delta T = RT^2 x(\mathrm{A})/\Delta H, \tag{3}$$

where R, T, ΔH, and $x(\mathrm{A})$ are the molar gas constant, the melting temperature, the enthalpy of fusion of water, and the molar fraction of impurity component A, respectively.

In this work, correction for the impurity effect was not conducted. The impurity effect of each element was calculated using Eq. 3 and taken as an uncertainty. The dominant elements are Si, B, and Na, and their depression effects are calculated to be 1.4 μK, 1.3 μK, and 0.6 μK, respectively. The total depression of the thirteen detected elements, evaluated as the sum of the effects of each element, is 3.3 μK. When the undetected elements, namely those whose molar values were possibly lower than the respective detecting limits, are taken into account by relying on the detection limits provided by the analyzer, their total effect does not exceed 3.5 μK. The molar values of the impurities and thus their depression effects should be doubled, considering

Table 3 Results of chemical impurity analysis (concentration in $\text{ng} \cdot \text{ml}^{-1}$)

Element	Concentration	Element	Concentration	Element	Concentration
Li	<0.05	Rb	<0.05	Eu	<0.05
Be	<0.05	Sr	<0.05	Gd	<0.05
B	7.4	Y	<0.05	Tb	<0.05
Na	7.4	Zr	<0.05	Dy	<0.05
Mg	<0.05	Nb	<0.05	Ho	<0.05
Al	0.2	Mo	0.09	Er	<0.05
Si	21	Ru	<0.05	Tm	<0.05
K	0.1	Rh	<0.05	Yb	<0.05
Ca	0.09	Pd	<0.05	Lu	<0.05
Sc	<0.05	Ag	<0.05	Hf	<0.05
Ti	<0.05	Cd	<0.05	Ta	<0.05
V	<0.05	In	<0.05	W	<0.05
Cr	0.07	Sn	<0.05	Re	<0.05
Mn	<0.05	Sb	<0.05	Ir	<0.05
Fe	0.1	Te	<0.05	Pt	<0.05
Co	<0.05	Cs	<0.05	Au	<0.05
Ni	0.3	Ba	0.06	TI	<0.05
Cu	0.5	La	<0.05	Pb	<0.05
Zn	0.2	Ce	<0.05	Bi	<0.05
Ga	<0.05	Pr	<0.05	Th	<0.05
Ge	<0.05	Nd	<0.05	U	<0.05
As	<0.05	Sm	<0.05		

that counter ions of these elements may exist in the water. Moreover, when the triple point of water is realized in the cells, where water and ice coexist, it is possible that the purity of the water is lower than that of the ice, since impurities are rejected into the liquid phase as water becomes pure ice when the ice mantle is prepared. Doubling the depression effect results in a value of 14 μK. Based on this analysis, we supposed that this uncertainty might be a reasonable value for our cells. However, it seems that there is still not enough evidence to claim this as a reliable uncertainty. For example, some impurities giving a significant potential effect, such as Hg, were not provided, and the uncertainty of the chemical analysis has not been adequately discussed. This situation leads us to estimate the standard uncertainty of the chemical impurities to be 20 μK.

3.3 Residual Gas Pressure

The internal gas pressure arising from gaseous impurities is one of the factors influencing the temperature realized by a water-triple-point cell, since it is impossible to remove residual gases completely during the manufacture of the cell. Cell Nos. 3, 5, 6, and 7 have an extended glass tube (inner diameter 5 mm, length 25 mm), which can be used as a McLeod gauge. The diameter of the trapped gaseous bubble of each cell is no more than 1 mm, and the temperature error is evaluated as less than 1 μK [4].

4 Reproducibility among TPW Cells

4.1 Measurement Procedure

Comparison measurements were performed to evaluate the reproducibility of the realized temperatures of the cells. The measurement system consists of a DC resistance bridge (Isotech TTI3), a standard platinum resistance thermometer (SPRT, Chino R800-2, 25 Ω at TPW), and a standard resistor (Tinsley 5685A, 10 Ω). The applied currents were 1 mA and 1.41 mA and the resistance ratio at 0 mA was calculated to correct the self-heating of the SPRT.

Cell No.4 was compared with the other six cells and treated as a reference for the comparison measurements since the isotopic composition of the cell is very close to that of V-SMOW.

In comparing cell Nos. 1 and 2 with cell No. 4, the order of the measurements was as follows: (1) cell No. 4, (2) cell No. 1, (3) cell No. 4, (4) cell No. 2, and (5) cell No. 4. The temperature difference between cell Nos. 1 and 4 was determined by the difference between the measured value at step (2) and the average of those at steps (1) and (3), to cancel the drift of the measurement system. The difference between cells No. 2 and No. 4 was obtained from the measurements at steps (3), (4), and (5). These measurements were performed in one day, and were repeated for 10 days using the same ice mantles. The average of these 10 measurements is treated as the temperature difference for a single ice mantle.

4.2 Results of Measurements

The comparison results are shown as the temperature differences from cell No. 4 in Fig. 2. Considering the depth of the well below the water surface of each cell, the hydrostatic pressure correction has been applied to the data in this figure. One plot represents the average of ten measurements for a single ice mantle.

The repeatability of the measurements for a single ice mantle was evaluated from the experimental standard deviation of ten measurements of the temperature difference to be typically 11 μK.

The comparison was performed for at least two ice mantles for each cell. The standard uncertainties of the reproducibility for different ice mantles for each cell were determined from the standard deviation of the measurements, the degrees of freedom, and the t-distribution function. The maximum value is 10.6 μK for cell No. 3, and the minimum is 2.6 μK for cell No. 1. The average, excluding cell No. 4, is 5.5 μK, and this is regarded as the uncertainty of the reproducibility for different ice mantles.

Figure 3 represents the temperature differences among cells after applying the isotopic correction to the averages of each cell shown in Fig. 2. All values are represented as differences from the mean of the seven cells. The error bars represent the reproducibility for different mantles ($k = 2$), as described above. In the case of cell No. 4, the average, 5.5 μK, is applied. It is clear that the seven cells show good agree-

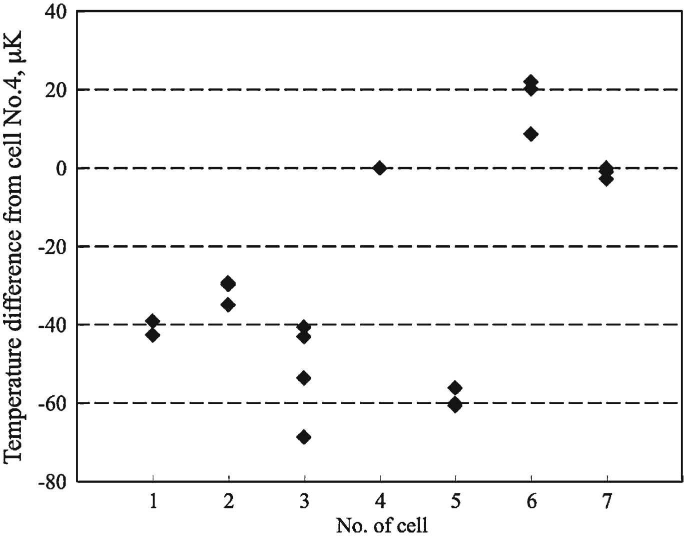

Fig. 2 Temperature differences among cells

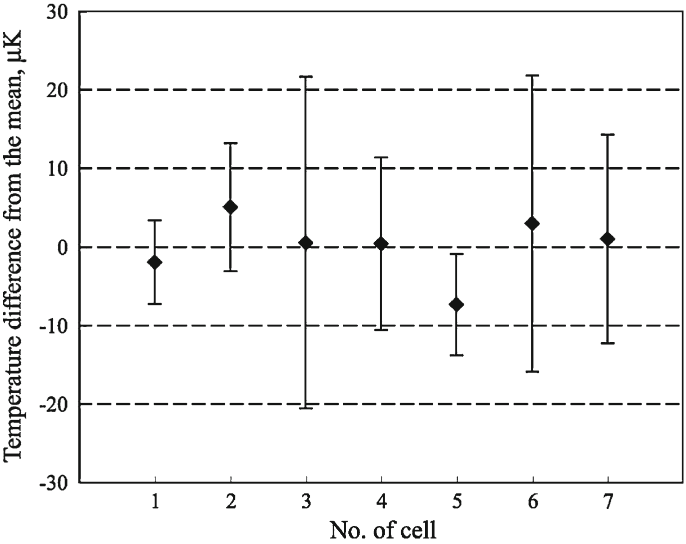

Fig. 3 Temperature differences among cells after applying the isotopic correction

ment. The standard deviation is 3.9 μK, and this is the standard uncertainty of the reproducibility for different cells.

5 Total Uncertainty of TPW Realization at NMIJ

The ambiguity of the definition of the isotopic composition of the triple point of water was reduced when the isotopic composition was strictly defined as V-SMOW in the

Table 4 Uncertainty budget of the new national reference of NMIJ

Source of uncertainty		u/μK ($k = 1$)	Subtotal
Ambiguity of the definition			
Isotopic fractionation effect			4.4
Isotopic correction			1.9
Repeatability of isotopic measurements	D	0.31	
	^{18}O	0.03	
Reproducibility among laboratories	D	1.10	
	^{18}O	0.10	
Systematic error of measurements	D	0.40	
	^{18}O	0.03	
Correcting equation	D	1.40	
	^{18}O	0.50	
Chemical impurities			20
Residual gas pressure in cell			1.0
Reproducibility			13
Repeatability for a single ice mantle		11.0	
Reproducibility for different ice mantles		5.5	
Reproducibility for different cells		3.9	
Combined standard uncertainty			24
Expanded uncertainty ($k = 2$)			49

revised SI. However, there is still ambiguity caused by the isotopic fractionation effect. In theory, the temperature differences between liquidus and solidus for D, ^{18}O, and ^{17}O are 13.3 μK, 1.9 μK, and 0.1 μK, respectively [10]. The sum of them, 15.3 μK, should be inherently counted in the uncertainty of the temperature of the triple point of water. The probability distribution is assumed to be rectangular with a width of 15.3 μK and a standard uncertainty of 4.4 μK.

The uncertainty budget shown in Table 4 summarizes the uncertainty components described above. The combined standard uncertainty ($k = 1$) is 24.3 μK, and the expanded uncertainty ($k = 2$) is 49 μK. This is the uncertainty of the new national reference of NMIJ.

6 Discussion

The previous national reference of the triple point of water reported in CCT-K7 was compared with the new national reference. In practice, the water-triple-point cell, No. TR0201, which was the national reference cell at the time of the CCT-K7, was compared with the new cells, Nos. 4 and 7, following the procedure described in the protocol of the CCT-K7. Figure 4 shows the results of the comparison. The previous reference is 42 μK lower than the new national reference, which is the mean of the seven cells. The error bar associated with the previous reference represents the expanded uncertainty, 80 μK, when a cell is calibrated by comparison with the new reference. This uncertainty consists of the uncertainty of the new national reference and that of the comparison measurements. The standard uncertainty of the comparison measurements

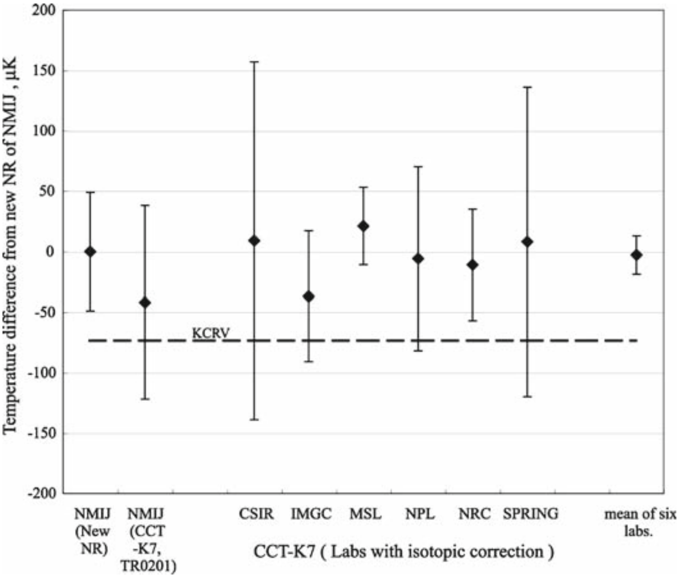

Fig. 4 Temperature differences of the previous national reference (NR) of NMIJ, and of those laboratories for which an isotopic correction can be applied to the CCT-K7 values, with respect to the new NR of NMIJ. Error bars represent expanded uncertainties ($k = 2$). NMIJ (New NR): the uncertainty of realization of temperature NMIJ (CCT-K7, TR0201): the uncertainty of calibration compared with new NR CCT-K7: the uncertainty from the CCT-K7 report Mean of six labs: deviation of six laboratories

is estimated to be 32 μK, and this includes the uncertainty of the reproducibility of the measurements, perturbing heat exchanges, correction of the hydrostatic head, and bridge noise.

The expanded uncertainty of the national reference has decreased from 302 μK at the time of CCT-K7 to 80 μK. Both the temperature difference with respect to the previous national reference, 42 μK, and the expanded uncertainty of the new national reference, 80 μK, are completely within the previous uncertainty. The new reliable information regarding isotopic composition and the chemical impurities has made the uncertainty smaller and shown the validity of the estimation of the previous uncertainty.

The KCRV of CCT-K7 and the values of the six laboratories that applied isotopic corrections are also shown in this figure. It is clear that the new reference of NMIJ and those of the six national references show good consistency, and that the validity of the manufacture of the cells, the correction of the isotopic composition, the measurements, and the estimation of the uncertainty is confirmed.

7 Conclusion

To improve the reliability of the TPW realization temperature of the national reference of NMIJ and to reduce its uncertainty, the isotopic compositions of seven water-triple-point cells were analyzed and the uncertainty of the isotopic correction was estimated. Chemical impurities and residual gases of the national reference cells were also analyzed. The realized temperatures of the seven cells were compared to each other after applying the isotopic correction.

Based on the obtained results, the expanded uncertainty of the new national reference of NMIJ is estimated to be 49 μK and the expanded uncertainty in calibrating a water-triple-point cell is 80 μK. The new reference was also compared with the previous national reference, which had an expanded uncertainty of 302 μK. The previous reference temperature is 42 μK lower than the new one. The new reference value is within the uncertainty of the previous national reference, and the new uncertainty is completely within the previous uncertainty. Furthermore, the new reference of NMIJ shows good agreement with the national references of the six laboratories reporting isotopic compositions in the CCT-K7. These facts confirm the validity and the linkage to the CCT-K7 of both the previous and the new national references of NMIJ.

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