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# Subsequent Bilateral Comparison to CCT-K3
# CIPM Key comparison CCT-K3.1

## Comparison of Standard Platinum Resistance Thermometers at the Triple Point of Water ($T$ = 273.16 K) and at the Melting Point of Gallium ($T$ = 302.9146 K)

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### ABSTRACT

A comparison of standard platinum resistance thermometers (SPRTs) has been carried out between the BIPM and the Laboratoire commun de métrologie LNE-CNAM using the melting point of gallium and the triple point of water. The temperature difference at Ga between the BIPM and the LNE-CNAM was determined as 108 µK with an associated combined standard uncertainty of 223 µK. This outcome indicates a present temperature difference of the BIPM of −65 µK with respect to an Average Reference Value from an earlier comparison, with an associated uncertainty of $u_c$ = 262 µK. It should be noted that the present BIPM scale is not independent, but traceable to cells used in KC7. The comparison results validate the re-establishment of thermometer calibrations at the BIPM for internal use.

## 1. INTRODUCTION

After an interruption in the calibration activity of standard platinum reference thermometers (SPRTs) at the International Bureau of Weights and Measures (BIPM) since 2006, the service is now being re-established exclusively for BIPM thermometers. The objective of the present comparison with the Laboratoire commun de métrologie LNE-CNAM (named LNE-CNAM in the report), carried out in September and October 2009, is to verify the equivalence and hence the quality of SPRT calibrations at the BIPM. An international comparison of the realization of the ITS-90 [1, 2], labelled as CCT-K3 in the framework of CIPM key comparisons in which the BIPM took part, was reported in 2002 [3, 4]. The comparison reported here is based on [3, 4] but with a limited scope, and a separate protocol was established [5].

The BIPM thermometer calibration is restrained to two fixed points of the international temperature scale ITS-90: the water triple point and the gallium melting point. The measurements have been made using the existing experimental equipment that has been serviced and monitored prior to use. Two thermometers belonging to the BIPM, ThA and ThB, were chosen as transfer instruments.

The result is represented by the temperature difference $\Delta T$ attributed to a measurement at the gallium melting point, and its associated uncertainty [3, 4], in form of

$$\Delta T_{\mathrm{BIPM-LNE}} = \frac{\frac{1}{2}(W_{\mathrm{BIPM,ThA}} - W_{\mathrm{LNE,ThA}}) + \frac{1}{2}(W_{\mathrm{BIPM,ThB}} - W_{\mathrm{LNE,ThB}})}{dW_{\mathrm{r}} / dT} . \tag{1}$$

The denominator $\mathrm{d}W_{\mathrm{r}}/\mathrm{d}T$ refers to the reference function [1, 2]. In the present case, $\mathrm{d}W_{\mathrm{r}}/\mathrm{d}T = 0.003\ 952$. The associated uncertainty is

$$u_{\mathrm{c}}(\Delta T_{\mathrm{BIPM-LNE}}) = \sqrt{\frac{s^2_{\mathrm{A_{BIPM}}}}{4n_{\mathrm{BIPM,ThA}}} + \frac{s^2_{\mathrm{A_{LNE}}}}{4n_{\mathrm{LNE,ThA}}} + s^2_{\mathrm{B_{BIPM}}} + \frac{s^2_{\mathrm{A_{BIPM}}}}{4n_{\mathrm{BIPM,ThB}}} + \frac{s^2_{\mathrm{A_{LNE}}}}{4n_{\mathrm{LNE,ThB}}} + s^2_{\mathrm{B_{LNE}}} + \frac{u^2_{\mathrm{ThA}}}{4} + \frac{u^2_{\mathrm{ThB}}}{4}} , \tag{2}$$

where $S_{\mathrm{A}}$ and $S_{\mathrm{B}}$ represent the type A and type B uncertainties, and $n$ the number of measurements [4]. The uncertainty components $u_{\mathrm{ThA}}$ *and* $u_{\mathrm{ThB}} \neq 0$ if a significant change in the thermometer response is observed after transport [3, 4].

The individual results of each laboratory were first communicated to the Executive Secretary of the Consultative Committee for Thermometry (CCT) before the laboratories had access to each other's results. The individual results of the BIPM and the LNE-CNAM and the final comparison results are reported here.

## 2. REALIZATION

Two long-stem SPRTs of the BIPM were selected as transfer instruments, each being identified in Table 1.

**Table 1.** Model and dimensions of the two SPRTs used as transfer instruments.

| Manufacturer | Model | Serial number | Length/mm | Diameter/mm |
|---|---|---|---|---|
| Leeds and Northrup[1] | 8167 | #439 | 465 | 7.3 |
| Leeds and Northrup | 8167 | #442 | 465 | 7.3 |

### 2.1. Measurements at the BIPM

The thermometers were first calibrated at the BIPM. The calibration consists of sequential measurements, each one including four cycles, using four different standards: a first water triple point cell (WTPC), a first gallium cell, a second gallium cell and a second WTPC. The measurements were performed using the internal BIPM technical procedures [6], applying a seven days annealing time to the ice mantles. The thermometers were then transported to the LNE-CNAM for calibration. Once back at the BIPM, the calibration sequence described above was repeated.

The apparatus used in the comparison consists of one high precision resistance bridge (ASL F18) to which a calibrated standard resistor of nominal resistance 25 Ω is connected. The normal measurement current is 1 mA. The reference resistor is maintained in a temperature-regulated oil bath at 23 °C. Two gallium cells were used and a heating rod was employed when preparing one of the cells. Five water triple point cells were used, prepared using $CO_2$ pellets at −78 °C (dry ice) and placed in a temperature-regulated water bath. The measurement series were carried out using computerized routines, and a numerical data sheet was used for the data analysis.

Once the thermometers were back at the BIPM, a test for the presence of moisture was made for both thermometers, using dry ice to cool the upper end of the SPRTs.

### 2.2. BIPM reference standards

Two cells were used as gallium reference standards: Ga24 from Pyro-Contrôle Chauvin Arnoult (manufactured with a LNE-CNAM licence) and a second, labelled Ga384 made by Isotech. These were corrected for hydrostatic effects. A heating rod was used when preparing the Ga24 cell.

Of the BIPM set of five water triple point cells, two were chosen as reference WTPCs: ASMW131 and ISO287, of which the ASMW131 was previously used as one of the BIPM references for the international key comparison CCT-K7 [7]. These were corrected for hydrostatic effects and for the shift induced by the isotopic compositions.

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[1] The name of the manufacturer is given in this paper only for reasons of technical information.

### 2.3. Measurements at the LNE-CNAM

The thermometers were calibrated at the LNE-CNAM following the LNE-CNAM internal technical procedure [8]. The number of measurements was limited to 3 cycles.

The apparatus used in the comparison consists of one high precision resistance bridge (ASL F900) to which a calibrated standard resistor of nominal resistance 100 Ω is connected. The normal measurement current is 2 mA. The reference resistor is maintained in a temperature oil bath at 25 °C.

### 2.4. LNE-CNAM reference standards

The French national temperature reference $\overline{T}_{\text{LNE-CNAM}}$ is based on the mean of the temperatures realized by a batch of $N$ cells $T_{\text{LNE-CNAM}}(i)$ :

$$\overline{T}_{\text{LNE-CNAM}} = \frac{\sum_{i=1}^{N} T_{\text{LNE-CNAM}}(i)}{N} = T_{\text{ITS-90}} \,. \tag{3}$$

A correction $C_{\text{LNE-CNAM}}(i)$ is calculated for each cell:

$$C_{\text{LNE-CNAM}}(i) = \overline{T}_{\text{LNE-CNAM}} - T_{\text{LNE-CNAM}}(i) \,. \tag{4}$$

For this comparison, one triple point cell (Hart Scientific SN 1020) and one gallium cell (Pyr 136) cell were chosen among the LNE-CNAM cells, for which the corrections are

$$\overline{T}_{\text{LNE-CNAM}} - T_{1020} = -17\,\mu\text{K} \tag{5}$$

for the triple point of water, and

$$\overline{T}_{\text{LNE-CNAM}} - T_{\text{Pyr}\,136} = 1\,\mu\text{K} \tag{6}$$

for the melting point of gallium.

# 3. RESULTS

## 3.1. BIPM results

### *3.1.1. Moisture test*

A test was made to detect any possible presence of moisture in the thermometers by inserting each thermometer into a WTPC. The resistance of the thermometer was recorded before and after using dry ice to cool the upper end of the SPRT sheath. The change in the triple point resistance was as large as 180 μK for the SPRT #439, whereas it was less than 20 μK for #442.

### *3.1.2. Heat flux test*

A heat flux test was made by recording the bridge reading of the thermometer inserted into a water triple point cell in a normal measurement configuration. The equipment was then isolated by using a large sheet of aluminized plastic film to thermally isolate the upper part of the thermometer and the connecting cable. The effect observed was less than 10 μK.

It was also made by comparing the measured hydrostatic pressure effect with the recommended parameters given by ITS-90. The effect was measured using a thermometer inserted into the water triple point cells, and successively changing the vertical position of the sensor of the thermometer in the dwell for each temperature measurement. The results are shown in Figure 1. The uncertainty of the effect is calculated according to [9] to be around 27 μK for both cells.

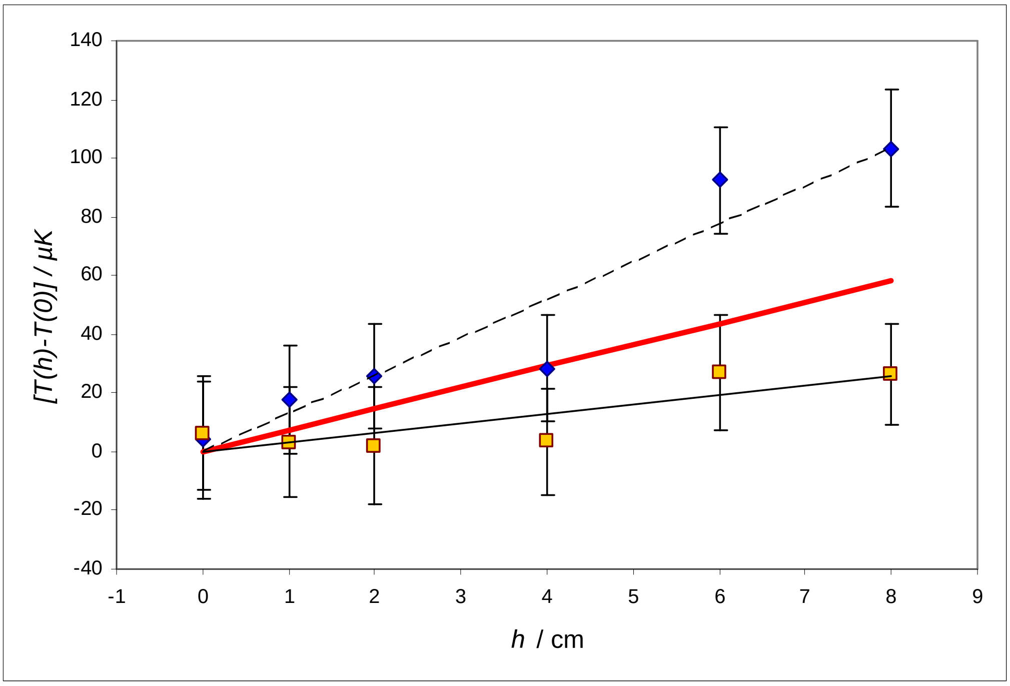

**Figure 1.** Measured temperature differences for different immersion depths in respect to the deepest position of the SPRT. The (blue) diamonds represent values obtained using the WTPC ASMW131; the (orange) squares represent the WTPC ISO287. The dashed and plain (black) curves show the fitted curves for the ASMW131 and ISO237 cells, respectively. The thick (red) curve represents the recommended value 0.73 mK/m [2].

### *3.1.3. Calibration data*

The data were corrected for the effect of the immersion depth due to the hydrostatic pressure using the coefficients recommended by ITS-90 [2]. The measured data given at zero current and corrected for the immersion depth are listed in Table 2, where $W_{\mathrm{BIPM}}$ is defined as

$$W_{\mathrm{BIPM}} = \frac{R(\mathrm{Ga24}) + R(\mathrm{Ga384})}{R(\mathrm{ASMW131}) + R(\mathrm{ISO287})}\,. \tag{7}$$

**Table 2.** Measured BIPM data used to calculate $W_{\mathrm{BIPM}}(\mathrm{Ga})$ for two SPRTs: #439 and #442. The standard uncertainty in the last digit is within parenthesis. These data are corrected for the immersion depth but not the isotopic effect.

| DD/MM/YYYY | #SPRT | $R$(ASMW131)/Ω | $R$(ISO287)/Ω | $R$(Ga24)/Ω | $R$(Ga384)/Ω | $W_{\mathrm{BIPM}}$ |
|---|---|---|---|---|---|---|
| 15/10/2009 | 439 | 25.560 183(1) | 25.560 194(2) | 28.578 302(1) | 28.578 281(1) | 1.118 078 3(1) |
| 16/10/2009 | 439 | 25.560 181(1) | 25.560 192(1) | 28.578 301(2) | 28.578 278(1) | 1.118 078 3(1) |
| 19/10/2009 | 439 | 25.560 185(2) | 25.560 188(2) | 28.578 311(2) | 28.578 270(5) | 1.118 078 3(1) |
| 20/10/2009 | 439 | 25.560 180(1) | 25.560 186(1) | 28.578 299(1) | 28.578 269(7) | 1.118 078 2(2) |
| 22/10/2009 | 439 | 25.560 178(2) | 25.560 190(2) | 28.578 292(2) | 28.578 276(4) | 1.118 078 2(1) |
| 26/10/2009 | 439 | 25.560 185(5) | 25.560 189(1) | 28.578 288(1) | 28.578 280(4) | 1.118 078 1(2) |
| 28/10/2009 | 439 | 25.560 189(2) | 25.560 190(2) | 28.578 291(2) | 28.578 278(3) | 1.118 078 0(1) |
| | | | | | | |
| 16/10/2009 | 442 | 25.557 481(1) | 25.557 489(1) | 28.575 083(1) | 28.575 073(1) | 1.118 070 8(1) |
| 19/10/2009 | 442 | 25.557 478(1) | 25.557 488(2) | 28.575 096(1) | 28.575 055(2) | 1.118 070 8(1) |
| 20/10/2009 | 442 | 25.557 488(1) | 25.557 490(2) | 28.575 084(1) | 28.575 067(4) | 1.118 070 5(1) |
| 22/10/2009 | 442 | 25.557 477(2) | 25.557 482(1) | 28.575 068(1) | 28.575 062(2) | 1.118 070 5(1) |
| 26/10/2009 | 442 | 25.557 463(3) | 25.557 468(1) | 28.575 052(1) | 28.575 050(3) | 1.118 070 6(1) |
| 28/10/2009 | 442 | 25.557 471(2) | 25.557 476(1) | 28.575 059(1) | 28.575 055(1) | 1.118 070 5(1) |

The isotopic compositions of the BIPM WTPCs are unknown. However, a correction for the isotopic composition of the water could be determined for the ASMW131 cell which had been used as a reference cell during the CCT-K7 comparison [7] of water triple point cells:

$$T_{\mathrm{V\text{-}SMOW}} - T_{\mathrm{ASMW131}} = 129\ \mu\mathrm{K}. \tag{8}$$

This correction is based on the measured temperature difference between the ASMW131 and a number of NMI cells for which the isotopic composition of the water was known. During the present comparison, the five BIPM WTPCs were compared on nine occasions. Assuming that the isotopic effect of ASMW131 has remained constant since the CCT-K7 comparison in 2003, the mean difference between $T_{\mathrm{ISO287}}$ and $T_{\mathrm{ASMW131}}$ is calculated to be 47 µK, which gives

$$T_{\mathrm{V\text{-}SMOW}} - T_{\mathrm{ISO287}} = 82\ \mu\mathrm{K}, \tag{9}$$

with an estimated uncertainty of 30 µK. The values of $W_{\mathrm{BIPM}}$, corrected for the isotopic effect, are shown in the graphs of Figures 2a and 2b.

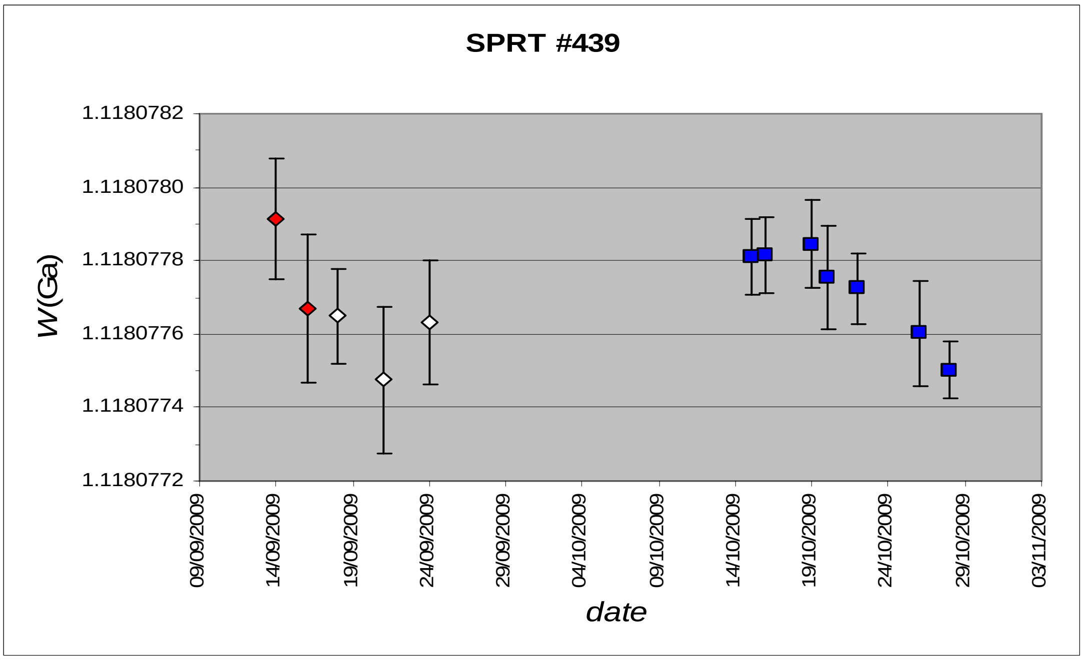

**Figure 2a.** Measured values of $W_{\mathrm{BIPM}}$ for the SPRT #439. The plain (red) diamonds represent values obtained using the WTPCs ASMW131 and KRISS1; the empty (white) diamonds and plain (blue) squares represent values obtained using the WTPCs ASMW131 and ISO287, respectively. The indicated uncertainty bars show the combined statistical uncertainty. No heating rod was used in the Ga24 cell for the data represented by diamonds.

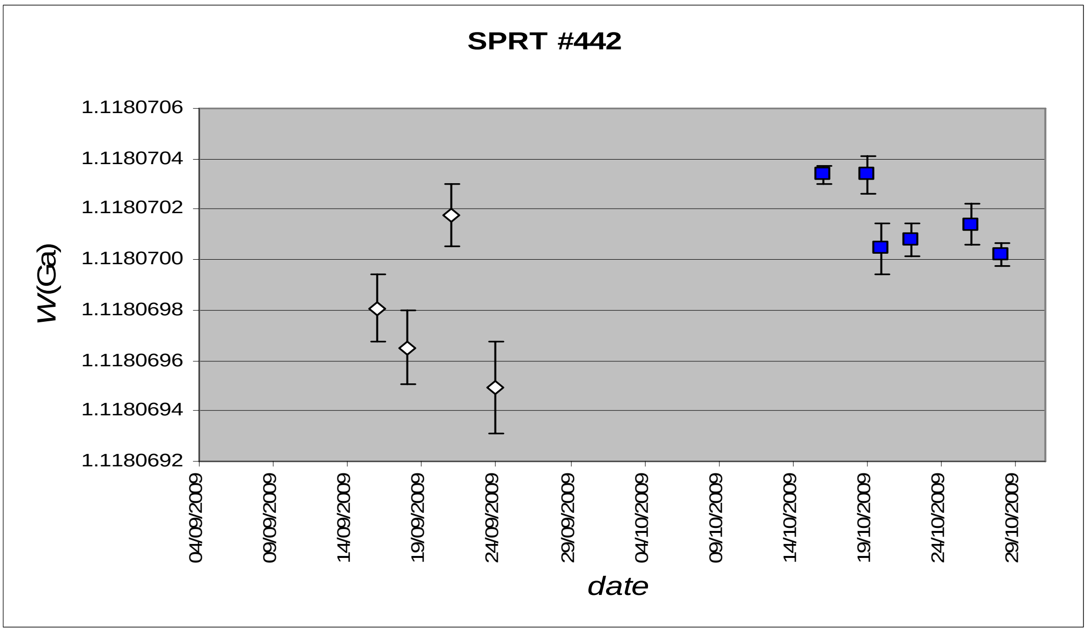

**Figure 2b.** Measured values of $W_{\mathrm{BIPM}}$ for the SPRT #442. The empty (white) diamonds and (blue) squares represent values obtained using the WTPCs ASMW131 and ISO287, respectively. The indicated uncertainty bars show the combined statistical uncertainty. No heating rod was used in the Ga24 cell for the data represented by diamonds.

The design of one of the water triple point cells, in combination with the length of the other cells, made it impossible to completely immerse the cells by the water in the water bath at the same time; water from the water bath otherwise entering the well. The upper part of the ice mantle was slightly diminished after two weeks of measurements. This may be one of the origins of the slight negative drift in $W$ as a function of time that can be observed for the blue squares in Figures 2a and 2b. The successive diffusion of impurities into the water around the thermometer well may be another contributing factor.

In the first week, before carrying the thermometers to the LNE-CNAM, the measurements were made without using a heating rod in the thermometer well of the Ga24 cell. However, a heating rod was constructed and used systematically to assure a liquid-solid interface in the Ga24 cell during the second week of measurements, after the thermometers were returned to the BIPM. As there is some doubt on the quality of the liquid-solid interface when not using the heating rod, it was decided prior to submitting the results to use only the data taken on the return of the thermometers to determine the comparison value.

The mean values of the $W_{\mathrm{BIPM}}$ data, including corrections for the immersion depths and the isotopic composition of the water, are listed in Table 3.

**Table 3.** Determined values of $W_{\mathrm{BIPM}}$ of the BIPM, the corresponding standard uncertainty of the mean value, and the estimated combined uncertainty based on the BIPM uncertainty budget.

| Thermometer | $W_{\mathrm{BIPM}}$ | $u_{\mathrm{A}}(W_{\mathrm{BIPM}})$ | $u_{\mathrm{c}}(W_{\mathrm{BIPM}})$ |
|---|---|---|---|
| #439 | 1.118 077 7 | 0.000 000 05 | 0.000 000 7 |
| #442 | 1.118 070 2 | 0.000 000 06 | 0.000 000 7 |

The measured mean difference in temperature between Ga24 and Ga384 is 130 µK, $u_{\mathrm{c}}$ = 80 µK.

## 3.2. LNE-CNAM Results

### *3.2.1. Heat flux test*

The LNE-CNAM heat flux test was made by comparing the measured hydrostatic pressure effect with the recommended parameters given by ITS-90. The effect was measured using a thermometer inserted into one of the water triple point cells, and successively changing the vertical position of the sensor of the thermometer in the dwell for each temperature measurement. This is illustrated by the graph in Figure 3. The effect is estimated from the graph to be less than 10 µK, but an uncertainty of 29 µK has been attributed for typical cases.

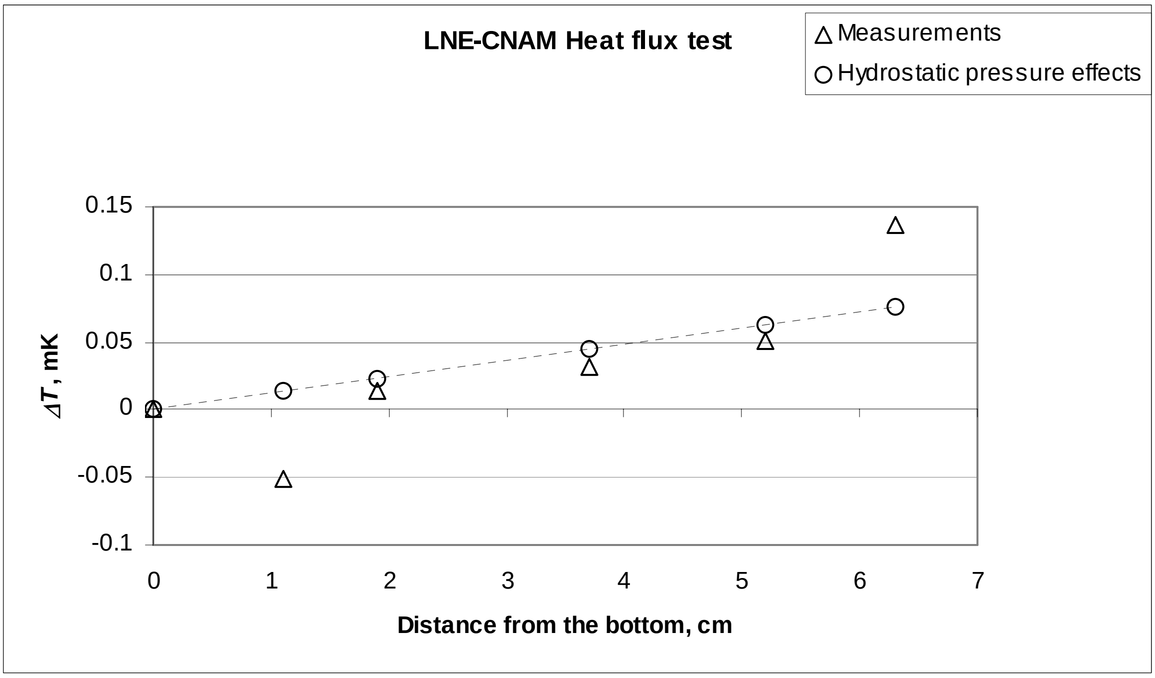

**Figure 3.** The LNE-CNAM heat flux test made by comparing the measured hydrostatic pressure effect with the recommended. Circular symbols represent the calculated values using the ITS-90 recommendation, while the triangular symbols represent measured data.

### *3.2.2. Calibration data*

The measured data given at zero current are listed in Table 4, where $W_{\text{LNE-CNAM}}$ is defined as

$$W_{\text{LNE-CNAM}} = \frac{R(\text{Ga})}{R(\text{WTP})}, \tag{10}$$

where $R$(Ga) is the resistance of the SPRT at the gallium fixed point and $R$(WTP) is the resistance in the WTP cell obtained after the measurement of $R$(Ga). The values $R$(WTP) are corrected for hydrostatic head and the isotopic composition. An appropriate individual correction is added. The values of $R$(Ga) are corrected for hydrostatic head and include also an individual correction (see Section 4). The determined values of $W$ and the uncertainties for LNE-CNAM are listed in Table 5.

**Table 4.** Measured LNE-CNAM data used to calculate $W_{\text{INM}}$ for two SPRTs: #439 and #442.

| DD/MM/YYYY | #SPRT | Fixed point | $R$(Ga)/Ω | $W_{\text{LNE-CNAM}}$ |
|---|---|---|---|---|
| 5/10/2009 | 439 | WTP | 25.560 180 (8) | |
| 5/10/2009 | 439 | Ga | 28.578 267 (3) | 1.118 077 5(0) |
| 6/10/2009 | 439 | WTP | 25.560 184 (5) | |
| 6/10/2009 | 439 | Ga | 28.578 264 (9) | 1.118 077 3(8) |
| 8/10/2009 | 439 | WTP | 25.560 185 (1) | |
| 8//10/2009 | 439 | Ga | 28.578 267 (6) | 1.118 077 3(8) |
| 9/10/2009 | 439 | WTP | 25.560 187 (5) | |
| | | | | |
| 5/10/2009 | 442 | WTP | 25.557 495 (9) | |
| 5/10/2009 | 442 | Ga | 28.575 070 (4) | 1.118 070 1(2) |
| 6/10/2009 | 442 | WTP | 25.557 493 (9) | |
| 6/10/2009 | 442 | Ga | 28.575 025 (2) | 1.118 069 5(0) |
| 8/10/2009 | 442 | WTP | 25.557 467 (8) | |
| 8//10/2009 | 442 | Ga | 28.575 029 (9) | 1.118 069 2(6) |
| 9/10/2009 | 442 | WTP | 25.557 477 (3) | |

**Table 5.** Determined values of $W_{\text{LNE-CNAM}}$ of the LNE-CNAM and the estimated combined uncertainty based on the LNE-CNAM uncertainty budget.

| Thermometer | $W_{\text{LNE-CNAM}}$ | $u_c(W_{\text{LNE-CNAM}})$ |
|---|---|---|
| #439 | 1.118 077 42 | 0.000 000 4 |
| #442 | 1.118 069 63 | 0.000 000 6 |

## 3.3. Uncertainty budgets

The uncertainty due to annealing was estimated to be negligible, as the thermometers are only used at temperatures from 0 °C to 30 °C. Further, no uncertainty has been attributed to the transport – the thermometers were accompanied by the operators on each trip, and were carefully packed. No significant difference in temperature was detected when comparing the measurements made before and after the transport.

### *3.3.1. BIPM uncertainty budget*

The estimated uncertainty budget for the temperature measurements of the BIPM is detailed in Table 6 [10]. It can be noted that some uncertainty contributions are identical for the measurement of the triple point of water and the gallium melting point, and will therefore cancel when calculating the uncertainty for $W_{\mathrm{BIPM}}$. However, their influence on the total uncertainty is negligible. The uncertainty linked to the realization of the triple point of water is larger than previously reported by the BIPM, basing the estimated uncertainties on recent observations and also taking into account the limited experience of the staff.

**Table 6.** Estimated uncertainty budget for the fixed points for the BIPM.

| **Influencing factor** | ***u*(WTP) / µK** | ***u*(Ga) / µK** |
|---|---|---|
| Repeatability | 10 | 10 |
| total $u_A$ | 10 | 10 |
| uncertainty linked in particular to impurities and the evolution of the surrounding of the thermometer well | 35 | 35 |
| immersion depth correction (hydrostatic pressure) | 7 | 12 |
| fixed point realization | 100 | 80 |
| isotopic correction | 30 | NA[2] |
| heat flux | 30 | 30 |
| oxidation | 5 | 5 |
| precision of resistance bridge F18 | 9 | 9 |
| precision of standard resistor | 13 | 13 |
| stability of the standard resistor | 1 | 1 |
| self-heating correction | 5 | 5 |
| total $u_B$ | 116 | 95 |
| $u_c$ | 116 | 95 |

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[2] Not Applicable

### *3.3.2. LNE-CNAM uncertainty budget*

The value of $W_{\text{LNE-CNAM}}$ is determined according the following mathematical model

$$W_{\text{LNE-CNAM}} = (1 + D_{Rs/3} + D_{Rs/4}) \times \left[\frac{(X_t + C_{Xt/1} + C_{Xt/2} + C_{Xt/3} + C_{Xt/4} + C_{Xt/5} + C_{Xt/6} + C_{Xt/7})}{(X_{0.01^\circ C} + C_{X0.01/1} + C_{X0.01/2} + C_{X0.01/3} + C_{X0.01/4} + C_{X0.01/5} + C_{X0.01/6} + C_{X0.01/7})}\right] \tag{11}$$

where the *C* and *D* terms represent applied correction factors, $R_s$ represents the standard resistance value, and the subscripts "*t*" and "0.01°C" indicate the gallium melting point and the water triple point, respectively. A detailed description of the components is given in Appendix A. The estimated uncertainty for each thermometer is summarized in Table 7.

**Table 7.** Estimated uncertainty budget for $W_{\text{LNE-CNAM}}$ of the LNE-CNAM.(a) included in *W* scatter; (b) negligible; (c) included in $C_{\text{Ga/1}}$; (d) strongly correlated with $C_{Ga/5}$.

| Symbol | Effect | *u*[#439] / mK | *u*[#442] / mK |
|---|---|---|---|
| $X_{\text{Ga}}$ | Repeatability of readings, linked in particular to bridge noise and electromagnetic interferences | (a) | (a) |
| $C_{\text{Ga/1}}$ | Uncertainty linked to purity and gas pressure | 0.047 | 0.047 |
| $C_{\text{Ga/2}}$ | Uncertainty linked to the hydrostatic pressure correction | 0.004 | 0.004 |
| $C_{\text{Ga/3}}$ | Uncertainty linked to perturbing heat exchanges | 0.029 | 0.029 |
| $C_{\text{Ga/4}}$ | Uncertainty linked to the self-heating correction | 0.029 | 0.029 |
| $C_{\text{Ga/5}}$ | Uncertainty linked to the bridge linearity | 0.050 | 0.050 |
| $C_{\text{Ga/6}}$ | Uncertainty linked to ac/dc current | (b) | (b) |
| $C_{\text{Ga/7}}$ | Uncertainty linked to the gas pressure | (c) | (c) |
| $X_{0.01\ ^\circ\text{C}}$ | Repeatability of readings, linked in particular to bridge noise and electromagnetic interferences | (a) | (a) |
|  | Repeatability of the temperature realized by cell, linked in particular to the buoyancy effect, impact of crystal defects and crystal size | 0.028 | 0.028 |
|  | Short-term repeatability of calibrated SPRT (dependent e.g. on hysteresis effects and small plastic deformations of the wire) | 0.013 | 0.066 |
| $C_{0.01^\circ\text{C}/1}$ | Uncertainty linked to purity and isotopic composition | 0.023 | 0.023 |

(*continued…*)

(**Table 7** *continued*)

| Symbol | Effect | $u$[#439] / mK | $u$[#442] / mK |
|---|---|---|---|
| $C_{0.01°\mathrm{C}/2}$ | Uncertainty linked to the hydrostatic pressure correction | 0.002 | 0.003 |
| $C_{0.01°\mathrm{C}/3}$ | Uncertainty linked to perturbing heat exchanges | 0.033 | 0.033 |
| $C_{0.01°\mathrm{C}/4}$ | Uncertainty linked to the self-heating correction | 0.033 | 0.033 |
| $C_{0.01°\mathrm{C}/5}$ | Uncertainty linked to the bridge linearity | (d) | (d) |
| $C_{0.01°\mathrm{C}/6}$ | Uncertainty linked to ac/dc current | (b) | (b) |
| $C_{0.01°\mathrm{C}/7}$ | Uncertainty linked to internal insulation leakage | 0.011 | 0.011 |
| $D_{RS/1}$ | Uncertainty linked to the stability of $R_\mathrm{S}$ | (b) | (b) |
| $D_{RS/2}$ | Uncertainty linked to the temperature of $R_\mathrm{S}$ | 0.004 | 0.004 |
| $S_{W\mathrm{Ga}}$ | $W$ scatter | 0.04 | 0.07 |
| $\boldsymbol{u_\mathrm{c}(W_\mathrm{LNE\text{-}CNAM})}$ | | 0.10 | 0.14 |

### 3.4. Comparison results

The uncertainty component $u_{ThA}$ *and* $u_{ThB}$ of (2) is here estimated as 0, and (2) can hence be approximated as a simple function of the combined uncertainty of each laboratory:

$$u_\mathrm{c}(\Delta T_\mathrm{BIPM-LNE}) = \sqrt{u^2_\mathrm{c,BIPM} + \frac{(u_\mathrm{c,LNE,ThA} + u_\mathrm{c,LNE,ThB})^2}{4}}\,. \tag{12}$$

Applying (1) and (8) to the data presented in Tables 2 and 4,

$$\Delta T_\mathrm{BIPM-LNE\text{-}CNAM} = 108\ \mu\mathrm{K}, \tag{13}$$

where $u_\mathrm{c}(\Delta T_\mathrm{BIPM-LNE\text{-}CNAM}) = 223\ \mu\mathrm{K}$ is obtained.

## 4. DISCUSSION

### 4.1. The stability of the LNE-CNAM water triple point and gallium standards

In view of the length of time which has passed since the CCT-K3 comparison was made, it is first important to trace the stability of the LNE-CNAM standards.

#### *4.1.1. Track of the LNE-CNAM water triple point standards*

Until January 2007, the French national temperature reference was based on the mean of the temperatures realized by a batch of nine water triple point cells. No information on the isotopic composition and impurity contents of these cells is available. Hence, ITS-90 was realized by calculating the mean temperature, $\overline{T}_{\text{LNE-CNAM}}$, obtained for $N$ cells belonging to the batch:

$$\overline{T}_{\text{LNE-CNAM}} = \frac{\sum_{i=1}^{N} T_{\text{LNE-CNAM}}(i)}{N} = T_{\text{ITS-90}}\,, \tag{14-a}$$

where $T_{\text{LNE-CNAM}}(i)$ represents the temperature of a separate cell $I$ (cf. (3)) A correction $C_{\text{LNE-CNAM}}(i)$ was calculated for each cell (cf. (4)):

$$C_{\text{LNE-CNAM}}(i) = \overline{T}_{\text{LNE-CNAM}} - T_{\text{LNE-CNAM}}(i)\,. \tag{14-b}$$

The uncertainty $u(\overline{T}_{\text{LNE-CNAM}})$ of the realization of the fixed point was estimated by

$$u(\overline{T}_{\text{LNE-CNAM}}) = \frac{\max(T_{\text{LNE-CNAM}}(i)) - \min(T_{\text{LNE-CNAM}}(i))}{2\sqrt{3}}\,. \tag{15}$$

As a consequence of the application of the CIPM recommendation 2 (CI-2005) [11] the French reference is since January 2007 based on the mean temperature, $T_{\text{mean}}$, of two separate cells (Harts Scientific SN 1422 and SN 1020). The isotopic composition of the water in #1020 is very close the V-SMOW definition, $T(\#1020) - T_{\text{V-SMOW}} = -0.4$ µK, while for the #1422 cell $T(\#1422) - T_{\text{V-SMOW}} = 70$ µK. After comparison and applying the appropriate corrections, it appears that the difference between the temperatures materialized by the cells #1020 et #1422 is 34 µK. Hence, the temperature for each cell is given by $\overline{T}_{LNE\text{-}CNAM} - T_{1020} = -17\mu K$ and $\overline{T}_{LNE\text{-}CNAM} - T_{1422} = 17\mu K$, respectively.

In CCT-K7 [7], France used a WTPC labelled UME6. Before January 2007,

$$\overline{T}_{\text{LNE-CNAM}} - T_{\text{UME6}} = 21\ \mu\text{K}. \tag{16}$$

After January 2007,

$$\overline{T}_{\text{LNE-CNAM}} - T_{\text{UME6}} = 121\ \mu\text{K}. \tag{17}$$

One can conclude that the reference has increased by 100 µK after introducing the definition of V-SMOW [12].

### *4.1.2. Track of the LNE-CNAM Gallium fixed point*

The gallium cell Gal2 was used in CCT-K3, while the cell Pyr136 was used in CCT-K3.1. The cell Gal2 was also employed in the EUROMET.T-K3 comparison. The stability of the gallium cell Gal2 is demonstrated by the LNE-CNAM[3] results in EUROMET.T-K3 and in CCT-K3. The LNE-CNAM results reported in EUROMET.T-K3 are consistent with those obtained in CCT-K3, cf. Table 25 and Figure 39 of [13].

The cells Gal2 and Pyr136 were compared in February 2000 and in January 2008. The results are illustrated in Figure 4. These measurements gave $T$(Gal2) – $T$(Pyr136) = 138 µK in 2000, whereas $T$(Gal2) – $T$(Pyr136) = 83 µK in 2008. The difference between these results (55 µK) is largely within the estimated uncertainties associated with the comparison of two gallium cells (130 µK for $k$ = 2) [14, 15].

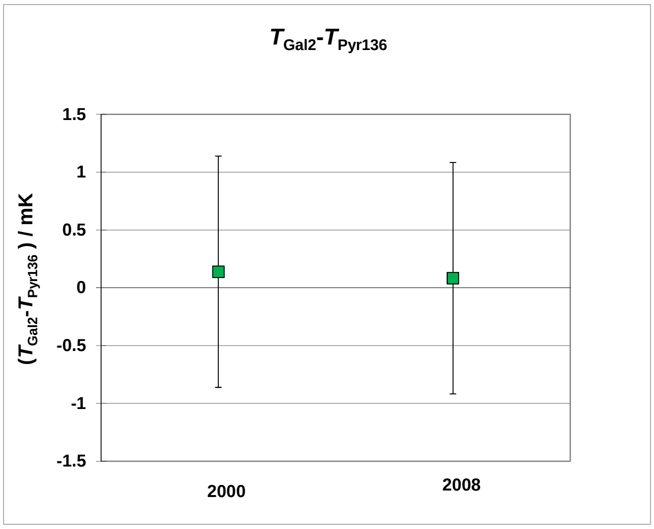

**Figure 4.** Internal comparison of the gallium cells Gal2 and Pyr136 in February 2000 and January 2008.

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[3] Previously BNM-INM

### 4.2. Comparison between the BIPM and the LNE-CNAM and traceability to CCT-K3

From the present comparison,

$$[W(\mathrm{Ga})\ \mathrm{BIPM}\ 2009] - [W(\mathrm{Ga})\ \mathrm{LNE}\ 2009]^{4} = +108\ \mu\mathrm{K}\ (u_{\mathrm{c}} = 223\ \mu\mathrm{K}) \tag{18}$$

is obtained (cf. (13)), where the LNE-CNAM reference cell in 2008 is Pyr136. The internal comparison of gallium cells at the LNE-CNAM gives

$$[T(\mathrm{Ga})\ \mathrm{LNE}\ 2009] - [T(\mathrm{Ga})\ \mathrm{LNE}\ 2000] = -55\ \mu\mathrm{K}\ (u_{\mathrm{c}} = 65\ \mu\mathrm{K}) \tag{19}$$

where the LNE-CNAM reference cell in 2000 is Gal2 (Fig. 4). Further, (16) and (17) give

$$[T(\mathrm{WTP})\ \mathrm{LNE}\ 2009] - [T(\mathrm{WTP})\ \mathrm{LNE}\ 2000] = +100\ \mu\mathrm{K}\ (u_{\mathrm{c}} = 65\ \mu\mathrm{K}). \tag{20}$$

From (19) and (20) it can be shown that

$$[W(\mathrm{Ga})\ \mathrm{LNE}\ 2009] - [W(\mathrm{Ga})\ \mathrm{LNE}\ 2000]^{4} = -163\ \mu\mathrm{K}\ (u_{\mathrm{c}} = 105\ \mu\mathrm{K}) \tag{21}$$

Combining (18) and (21),

$$[W(\mathrm{Ga})\ \mathrm{BIPM}\ 2009] - [W(\mathrm{Ga})\ \mathrm{LNE}\ 2000]^{4} = -\ 55\ \mu\mathrm{K}\ (u_{\mathrm{c}} = 246\ \mu\mathrm{K})^{5}. \tag{22}$$

As the work described in this paper is a key comparison, linked to the previous key comparison CCT-K3, it must also provide a value for the degrees of equivalence. However, no key comparison reference value was reported in the CCT-K3 comparison 2002 [3, 4]. Therefore, the results presented here are linked to the average reference value (ARV) value of $T$(Ga) created after the completion of CCT- K3 [16]. A similar approach is made in [17].

The CCT Working Group 8 addendum [16] to the Final report of CCT-K3 [3, 4] gives the comparison results of CCT-K3 relative to the average reference value ARV 2002:

$$[T(\mathrm{Ga})\ \mathrm{LNE}\ 2002] - [T(\mathrm{Ga})\ \mathrm{ARV}\ 2002] = -10\ \mu\mathrm{K}\ (u_{\mathrm{c}} = 155\ \mu\mathrm{K}). \tag{23}$$

Using the information of (22) and (23) one obtains

$$[T(\mathrm{Ga})\ \mathrm{BIPM}] - [T(\mathrm{Ga})\ \mathrm{ARV}\ 2002] = -\ 65\ \mu\mathrm{K}\ (u_{\mathrm{c}} = 262\ \mu\mathrm{K}). \tag{24}$$

---

[4] The difference in $W$ is here expressed in terms of temperature.

[5] The correlations have been disregarded here.

The associated uncertainty in (24) is based on the uncertainties of $W$(Ga) BIPM 2009, $W$(Ga) LNE 2009, $T$(ARV – LNE) 2002 and the uncertainty of the stability of [$W$(Ga) LNE 2009] − [$W$(Ga) LNE 2000], as indicated in Table 8.

**Table 8.** Contributions and calculation of the uncertainty of [$T$(Ga) BIPM] − [$T$(Ga) ARV 2002].

| **Contribution** | ***u* / µK** |
|---|---|
| WTP BIPM (cf. Table 6) including the propagation of uncertainty ($W = 1.118$) [9] | 130 |
| $T$(Ga) (cf. Table 6) | 95 |
| $W$(Ga) LNE-CNAM (cf. Table 7) | 120 |
| Stability of $W$(Ga) LNE-CNAM (cf. Eq. (19)) | 65 |
| $u$(ARV–{LNE-CNAM}) [16] | 155 |
| $\boldsymbol{u_c}$ | 262 |

The difference between the participating NMIs in CCT-K3 and the ARV in 2002 for $T$(Ga) is listed in Appendix B. The determined difference in 2009 for the BIPM relative to the ARV is also given.

In a former comparison of SPRTs [4], a temperature difference of

$$\Delta T_{\mathrm{BIPM-LNE\text{-}CNAM}}(\mathrm{Ga}) = -110\ \mu\mathrm{K}\ (u_c = 170\ \mu\mathrm{K}) \tag{25}$$

was measured for the melting point of gallium [4]. The two comparison results for $\Delta T_{\mathrm{BIPM-LNE\text{-}CNAM}}$(Ga) are represented in graphical form in Figure 5, illustrating that the present comparison result is consistent with the former result. It can be pointed out that neither the BIPM nor the LNE-CNAM applied a correction for the isotopic effect in the first comparison.

It should be noted that the present BIPM scale is not independent, but traceable to cells used in KC7. These results confirm that the BIPM has restored its capacity for temperature calibrations, while the LNE-CNAM has maintained its stated level of accuracy.

The rather large moisture effect identified for one of the thermometers does not seem to influence the reproducibility to any large extent, probably because the thermometer was used during a relatively short time period, and in a fairly narrow temperature interval. The evolution of this thermometer will be followed closely.

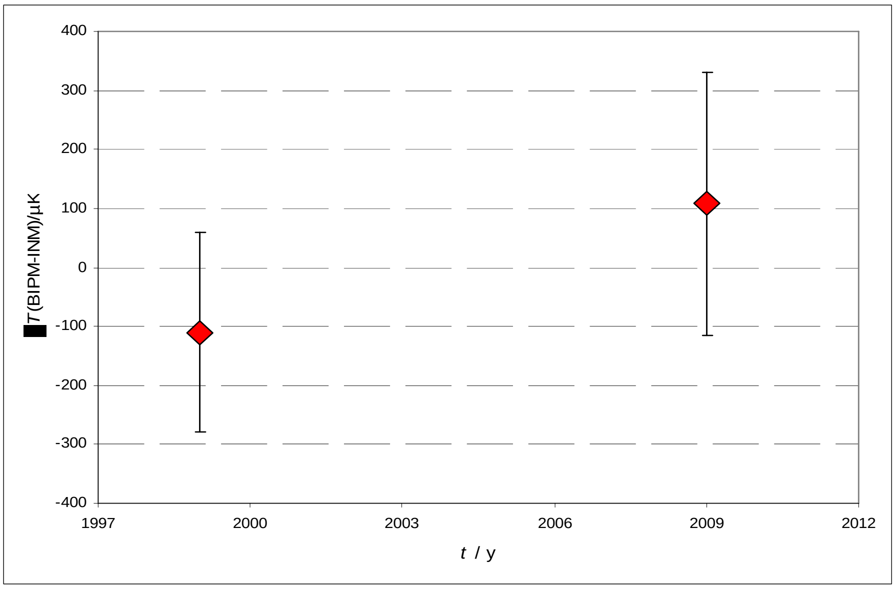

**Figure 5.** The comparison result of $\Delta T_{\mathrm{BIPM\ -\ LNE\text{-}CNAM}}$(Ga) of CCT-K3, carried out 1997-2001, and in 2009, respectively.

## 5. CONCLUSION

The results of the LNE-CNAM represented the median in the CCT comparison results of 2003, where the BIPM results were close to the median [4]. The bilateral comparison between the BIPM and the LNE-CNAM has allowed a determination of a present temperature difference of the BIPM with respect to the ARV of [$T$(Ga) BIPM] − [$T$(Ga) ARV 2002] = −65 µK ($u_c$ = 262 µK). The smallest calibration uncertainty required by the BIPM users is currently 300 µK. Hence the measured temperature difference and associated uncertainty resulting from this 2009 comparison fulfils the present BIPM requirements for the calibrations of the BIPM thermometers.

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# Appendix A

## Uncertainty components of the LNE-CNAM estimated uncertainty budget

The value of $W_{Ga}$ is determined according the following mathematical model obtained from the relationship.

$$W_{\mathrm{Ga}} = \frac{(R_s + C_{Rs/3} + C_{Rs/4}) * (X_t + C_{Xt/1} + C_{Xt/2} + C_{Xt/3} + C_{Xt/4} + C_{Xt/5} + C_{Xt/6} + C_{Xt/7})}{(R_s) * (X_{0.01\,^\circ C} + C_{X\,0.01/1} + C_{X\,0.01/2} + C_{X\,0.01/3} + C_{X\,0.01/4} + C_{X\,0.01/5} + C_{X\,0.01/6} + C_{X\,0.01/7})}$$

which also can be written as

$$W_{\mathrm{Ga}} = (1 + D_{Rs/3} + D_{Rs/4}) \cdot \frac{(X_t + C_{Xt/1} + C_{Xt/2} + C_{Xt/3} + C_{Xt/4} + C_{Xt/5} + C_{Xt/6} + C_{Xt/7})}{(X_{0.01^\circ C} + C_{X0.01/1} + C_{X0.01/2} + C_{X0.01/3} + C_{X0.01/4} + C_{X0.01/5} + C_{X0.01/6} + C_{X0.01/7})} \cdot$$

| | |
|---|---|
| $R\mathrm{s}$ | Reference resistor value at the time of TPW measurement |
| $D_{\mathrm{Rs/3}}$ | Relative drift of the resistance of the reference between TPW and FP measurements $= C_{Rs/3} / R\mathrm{s}$ |
| $D_{Rs/4}$ | Relative temperature variation of resistance of the reference between TPW and FP measurements $= C_{Rs/4} / R\mathrm{s}$ |

*Effects linked with triple point of water calibration*:

| | |
|---|---|
| $X_{0.01°C}$ | Reading on the bridge at the triple point of water |
| $C_{x0.01/1}$ | Water triple point reference including isotope variation |
| $C_{x0.01/2}$ | Hydrostatic pressure correction |
| $C_{x0.01/3}$ | Perturbing heat exchanges |
| $C_{x0.01/4}$ | Self-heating correction |
| $C_{x0.01/5}$ | Bridge linearity |
| $C_{x0.01/6}$ | AC/DC measurement correction |
| $C_{x0.01/7}$ | SPRT internal insulation leakage correction |

*Effects linked with the considered fixed point calibration*:

| | |
|---|---|
| $X_t$ | Reading on the bridge |
| $C_{Xt/1}$ | Chemical impurities |
| $C_{Xt/2}$ | Hydrostatic pressure correction |
| $C_{Xt/3}$ | Perturbing heat exchanges |
| $C_{Xt/4}$ | Self-heating correction |
| $C_{Xt/5}$ | Bridge measurement correction, lack of linearity |
| $C_{Xt/6}$ | AC/DC measurement correction |
| $C_{Xt/7}$ | Gas pressure correction |
| $S_{Wt}$ | $W_t$ scatter |

## Guide for components and method of estimation

| Quantity | Standard Uncertainty | Method |
|---|---|---|
| $X_t$ | Repeatability of readings. No change during a short time (bridge noise, electromagnetic interferences) | - Same SPRT<br>- Same cell<br>- Same freezing<br>- Same day |
| $C_{Xt/1}$ | Purity | - Quoted from the dispersion of a batch of cells. |
| $C_{Xt/2}$ | Hydrostatic pressure correction | Estimated from the uncertainty of the sensible element position and the uncertainty of the free liquid level |
| $C_{Xt/3}$ | Perturbing heat exchanges (between the sensor and the surrounding parts different in temperature from the liquid-solid phase change) | -Deviation from expected hydrostatic pressure correction obtained by changing immersion depth over 5 cm (length of the sensor)<br>-Modification of the thermal exchange between thermometer and its environment |
| $C_{Xt/4}$ | self-heating correction | Resolution of the bridge readings, uncertainty on the ratio between the two measuring currents<br>Variation in self heating correction observed in an apparent similar environment |
| $C_{Xt/5}$ | bridge linearity | Use of calibrated resistor and RBC for checking the bridge. Comparison between readings on different bridges. Checking the symmetry of the bridge ( R1/R2 = 1/(R2/R1) ?) |
| $C_{Xt/6}$ | Difference between AC and DC measurements | Estimated by using DC and AC bridge |
| $C_{Xt/7}$ | Gas pressure in the cell | Uncertainty on pressure value. |
| $X_{0.01°C}$ | a) Repeatability of readings. No change during a short time (bridge noise, electromagnetic interferences) | - Same SPRT<br>- Same cell and same mantle realization<br>- Same day |
|  | b) Repeatability of temperature realised by cell (buoyancy effect, impact of crystal defects, crystal size) | - Same SPRT (assumed stable)<br>- Same cell<br>- Different realisations of the mantle (1)<br>- Different dates of measurement for take into account mantle ageing (2) |
|  | c) Short Repeatability of SPRT to be calibrated (hysteresis effect, small plastic deformation of the wire) | - Same cell<br>- Variation between TPW measurement before and after the considered fixed point |

| Quantity | Standard Uncertainty | Method |
|---|---|---|
| $C_{X0.01/1}$ | Purity and isotopic composition | Comparison between several cells from different sources in the same conditions. Use of the interlaboratory comparison data.<br>Correction for isotopic composition Use of the isotopic analysis given by a CEA laboratory (with associated uncertainties). |
| $C_{X0.01/2}$ | Hydrostatic pressure correction | Estimated from the uncertainty on the distance between the platinum sensor and the free liquid level |
| $C_{X0.01/3}$ | Perturbing heat exchanges (between the sensor and the surrounding parts different in temperature from the liquid-solid phase change) | -Deviation from expected hydrostatic pressure correction obtained by changing immersion depth over 5 cm (length of the sensor)<br>-Modification of the thermal exchange between thermometer and its environment |
| $C_{X0.01/4}$ | self-heating correction | Resolution of the bridge readings, uncertainty on the ratio between the two measuring currents<br>Variation in self heating correction observed in an apparent similar environment |
| $C_{X0.01/5}$ | bridge linearity | Use of calibrated resistor and RBC for checking the bridge.<br>Comparison between readings on different bridges.<br>Checking the symmetry of the bridge<br>( R1/R2 = 1/(R2/R1) ?) |
| $C_{X0.01/6}$ | Difference between AC and DC measurements | Estimated by using DC and AC bridge |
| $C_{X0.01/7}$ | SPRT internal Insulation leakage (if any) | Decrease in resistance over some hours in the triple point |
| $D_{RS/3}$ | Lack of stability of the reference resistance value | Negligible if measurement performed in a short time (within two successive days) |
| $D_{RS/4}$ | Change in value of the standard resistor with thermostat temperature | - uncertainty on calibrating temperature<br>- uncertainty on temperature at time of use<br>- uncertainty on temperature coefficient |
| $S_{WGa}$ | $W_{Ga}$ scatter | - Same SPRT<br>- Same cell<br>- Different W values |

## Appendix B

## Temperature of the determined gallium melting point relative to the average reference value 2002

The determined temperature of the melting point at gallium of the CCT-K32002 is listed below. Also given are the comparison results issued by the BIPM and the LNE-CNAM in 2009.

| NMI[6] | [$T$(Ga) NMI] – [$T$(Ga) ARV] / µK | $u_c$ / µK |
|---|---|---|
| LNE-CNAM 2002 | 10 | 155 |
| IMGC 2002 | 130 | 70 |
| KRISS 2002 | 40 | 205 |
| MSL 2002 | 210 | 105 |
| NIM 2002 | -550 | 265 |
| NIST 2002 | 40 | 55 |
| NML[7] 2002 | -120 | 255 |
| NPL 2002 | -130 | 225 |
| NRC 2002 | -100 | 135 |
| NRLM 2002 | -330 | 40 |
| PTB 2002 | 240 | 135 |
| SMU 2002 | 80 | 115 |
| VNIIM 2002 | 50 | 125 |
| VSL 2002 | -190 | 215 |
| BIPM 2002 | -50 | 100 |
| BIPM 2009 | -65 | 262 |

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[6] For the NMI acronyms, cf. http://www.bipm.org

[7] Now NMIA.