BUREAU INTERNATIONAL
DES POIDS ET MESURES

Bilateral comparison of 10 kΩ standards
(ongoing BIPM key comparison BIPM.EM-K13.b)
between the NIMT (Thailand) and the BIPM

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1 Introduction

A comparison of values assigned to 10 kΩ resistance standards was carried out between the BIPM and the NIMT (Thailand) in the period January 2009 to April 2009.
Two 10 kΩ BIPM travelling standards (TEGAM, SR104 type) were calibrated first at the BIPM, then at the NIMT and again at the BIPM after their return. The measurement periods are referred to as:

'Before' measurements at the BIPM: January 2009
NIMT measurements: March 2009
'After' measurements at the BIPM: March 2009 - April 2009

The BIPM calibrations are corrected to the reference temperature 23.000 °C and the reference pressure 1013.25 hPa.
According to the protocol, the NIMT did not apply pressure and temperature corrections to its results. The corrections were made by the BIPM, using the temperature and pressure coefficients of the standards together with the temperature and pressure measurements provided by the NIMT.

The calibration reports provided by the NIMT are summarized by the BIPM in section 3 of the present report.

There is no evidence of a single linear drift of each standard over the whole period of the comparison (three measurement periods, 'Before', 'NIMT' and 'After': see Figures 1 and 2). Moreover, the two standards exhibited a significant increase (about 4 parts in 10^8) of their resistance after their return to the BIPM, and a subsequent decrease during about two weeks, down to a stable value. The values corresponding to this transient period (white squares on Figure 1 and Figure 2) have not been used in the calculation.

For each period, the calibration value assigned to each standard is the mean value of the measurements performed during this period, with an associated standard uncertainty.

The difference between the NIMT and the BIPM calibrations of a given standard R_i can be written as:

$$\Delta_i = R_{\mathrm{NIMT},\,i} - R_{\mathrm{BIPM},\,i}$$

If two standards are used, the mean of the differences is:

$$\Delta_{\mathrm{NIMT-BIPM}} = \frac{1}{2}\sum_{i=1}^{2}(R_{\mathrm{NIMT},\,i} - R_{\mathrm{BIPM},\,i}) \qquad (1)$$

This expression can also be written as:

$$\Delta_{\mathrm{NIMT-BIPM}} = \frac{1}{2}\sum_{i=1}^{2}R_{\mathrm{NIMT},\,i} - \frac{1}{2}\sum_{i=1}^{2}R_{\mathrm{BIPM},\,i} \qquad (2)$$

which is the difference of the means.

2 Measurements at the BIPM

2.1 BIPM calibrations

The BIPM measurements were carried out by comparison with a set of two 10 kΩ reference resistors (referred to as B10K1 and B10K2) whose values are known with respect to the BIPM quantized Hall resistance (QHR) standard. The comparison was performed using a Warshawsky bridge operating with a 0.1 mA DC current.
In order to minimize the interpolation and extrapolation uncertainty, the 10 kΩ reference was calibrated against the QHR in February 2009, during the first part of the comparison.

The 10 kΩ travelling standards were kept in a temperature-controlled air bath at a temperature which is close (less than 0.05 °C) to the reference temperature. The temperature of the standards was determined by means of a calibrated platinum resistance thermometer (SPRT), in conjunction with thermocouples.
The BIPM measurements are summarized in Table 2 and the uncertainty budget in Table 1.

Source of uncertainty	relative standard uncertainty / 10^{-9}
Imperfect realization of $R_H(2)$	2.0
Link $R_H(2)$ / 100 Ω	3.0
Link 100 Ω / 10 000 Ω	5.0
Link 10 000 Ω / (mean reference B10K1-B10K2)	7.0
Extrapolation of mean value of 10 kΩ reference	8.0
Measurement of the voltage applied to the bridge	5.0
Leakage resistances	5.0
Temperature correction for travelling standard	3.0
Pressure correction for travelling standard	2.0
Combined uncertainty u_2	**15 × 10^{-9}**

Table 1: BIPM uncertainty budget for the calibration of the 10 kΩ travelling standards.

BIPM	**Relative difference from nominal 10 kΩ value**			
Standard #	BEFORE / 10^{-6}	Std. dev. u_{1B} / 10^{-9}	AFTER / 10^{-6}	Std. dev. u_{1A} / 10^{-9}
B10K10	0.163	1.1	0.170	1.1
B10K12	0.510	0.9	0.494	0.7

Mean value of 'Before' and 'After'			
Standard #	**mean** / 10^{-6}	Exp. Std. dev. u_1 / 10^{-9}	Systematic u_2 / 10^{-9}
B10K10	**0.167**	1	15
B10K12	**0.502**	1	15

Table 2: Summary of the BIPM calibrations. The dispersion is estimated by the standard deviations of the mean, and 'systematic' refers to the sources of uncertainty that do not contribute to the variability of the results.

The value attributed to the *i*-th standard is the arithmetic mean of the "Before" and "After" values:

$$R_{\text{BIPM, i}} = (R_{\text{Before}, i} + R_{\text{After, i}})/2$$

For each standard, the uncertainty u_1 associated with the dispersion is the quadratic mean of the standard deviations "Before" and "After".

$$u^2_{1,i} = (u^2_{1\text{Before}, i} + u^2_{1\text{After}, i})/2^2$$

u_2 is the uncertainty arising from the combined contributions associated with the BIPM measurement facility and the traceability, as described in Table 1. This component is assumed to be strongly correlated between calibrations performed in the same period.

For a single standard, the BIPM uncertainty $u_{\mathrm{BIPM},\,i}$ is obtained from: $u^2_{\mathrm{BIPM},\,i} = u^2_{1,\,i} + u^2_{2,\,i}$

Unlike the $u_{1,\,i}$, the $u_{2,\,i}$ are assumed to be correlated.

Using expression (2), when the mean (for two standards) of the NIMT-BIPM relative difference is calculated, the BIPM contribution to the uncertainty is:

$$u^2_{\mathrm{BIPM}} = \sum_{i=1}^{2} \frac{u^2_{1,\,i}}{2^2} + u^2_2 \tag{3}$$

Using the values shown in Table 2, the relative standard uncertainty u_{BIPM} is

$$u_{\mathrm{BIPM}} = 15 \times 10^{-9}.$$

2.2 <u>Uncertainty associated with the transfer</u>

u_{d} is the uncertainty associated with the drift (or the step changes) of the travelling standards observed after their return to the BIPM.
As described in Section 1, the values observed during the transient period after the return have not been used. The measurement period 'After' starts on the 6 April 2009 (blue squares on Figure 1 and Figure 2).

The final resistance value attributed by the BIPM (the mean of the 'Before' and 'After' measurements) is in the middle of the step d: $d = \left|(R_{\mathrm{After}} - R_{\mathrm{Before}})\right|$

As we have no clear knowledge about the behaviour of the standards during the period between 'Before' and 'After', it is assumed that the actual resistance could have had any value in the range d, with equal probability.

Assuming a rectangular probability distribution, $u_{\mathrm{d}} = \frac{d}{2} \cdot \frac{1}{\sqrt{3}}$

Another source of uncertainty associated with the transfer would be a difference in the operating currents used by the two laboratories, influencing the resistance of the standards through their power coefficients.
For a single standard, the transfer uncertainty $u_{\mathrm{T},\,i}$ is obtained from: $u^2_{\mathrm{T},\,i} = u^2_{\mathrm{d},\,i} + u^2_{\mathrm{P},\,i}$

In the present case, the nominal operating current is 0.1 mA at both laboratories. The value of the relative standard uncertainty u_{P} associated with possible power effects is estimated to be negligible.
Following the same reasoning as in expression (3), the uncertainty u_{T} associated with the transfer (for the mean of two standards) is:

$$u^2_{\mathrm{T}} = \sum_{i=1}^{2} \frac{u^2_{\mathrm{d},\,i}}{2^2}$$

	Transfer
Standard #	Drift $u_{\mathrm{d}} / 10^{-9}$
B10K10	2
B10K12	4
Combined u_{T}	**2.2**

Table 3: Uncertainty u_{T} associated with the transfer.

3 Measurements at the NIMT

3.1 Method of calibration:

The travelling standards, placed in a temperature-controlled laboratory at (23 ± 2)°C, were measured by comparison with a 1000 Ω reference standard (Tinsley, 5685B immersed in an oil bath) using a Quantum Hall Resistance Bridge Model MI 6010 Q, repeatedly, in a four-terminal configuration. The temperature of the 1000 Ω reference standard (R_S) is estimated to coincide with the monitored temperature within 0.02°C.

The 1000 Ω reference standard is itself known in term of the recommended value of the von Klitzing constant, $R_{K\text{-}90}$ = 25 812.807 Ω. In order to minimize the interpolation and extrapolation uncertainty, this reference was calibrated against the QHR in February 2009.

3.2 Operating conditions:

Operating current: 0.1 mA dc.
Atmospheric pressure range: 1005.9 hPa to 1010.5 hPa.

3.3 NIMT results:

The travelling standards were measured 10 times in the period 2 March – 17 March 2009. The results are summarized in Table 4.

Serial No. of standard:	Resistance value (Ω)	Repeatability / 10^{-8}	Mean temperature / °C	Mean atmospheric pressure / hPa
B10K10	10 000.007 8	0.9	23.36	1 008.6
B10K12	10 000.011 9	0.9	23.33	1 007.7

Table 4: Summary of the NIMT calibrations.

The repeatability is estimated by the standard deviation of the mean of the series of 10 measurements.

The NIMT results are corrected to the reference temperature and the reference pressure using the coefficients shown in Table 5. The corrections are shown in Table 6.

	Relative temperature coefficients		Relative pressure coefficients.
Standard #	Alpha$_{23}$ / (10^{-6}/K)	Beta / (10^{-6}/K²)	/ (10^{-9}/hPa)
B10K10	– 0.040	– 0.022	– 0.314
B10K12	0.010	– 0.023	– 0.226

Table 5: Temperature and pressure coefficients of the travelling standards.

Reference temperature = 23.000°C Reference pressure = 1013.25 hPa		
	Corrections	
Standard #	For temperature	For pressure
B10K10	+ 1.7 x 10^{-8}	– 0.1 x10^{-8}
B10K12	– 0.1 x 10^{-8}	– 0.1 x 10^{-8}

Table 6: Corrections for temperature and pressure applied to the NIMT results.

The uncertainties on temperature and pressure measurements at the NIMT are 0.15 °C and 2.5 hPa respectively.
Taking into account the differences from the reference temperature and reference pressure, and the uncertainties associated with the coefficients, the uncertainties u_{Temp} and u_{Press} associated with the temperature and pressure corrections applied by the BIPM are estimated to be $u_{\mathrm{Temp}} = 2.0 \times 10^{-8}$ and $u_{\mathrm{Press}} = 0.3 \times 10^{-8}$, leading to a combined uncertainty $u_3 = 2.0 \times 10^{-8}$.

Source of uncertainty	Type	Relative standard uncertainty / 10^{-8}
Reference standard R_S uncertainty (a)	B	5.73
Drift of R_S	B	5.77
Linearity of ratio 10 000 / 1000	B	4.50
Ratio 10 000 / 1000	B	1.20
Resolution	B	0.029
Error of scanner	B	2.00
Oil temperature on R_S (b)	B	3.46
Combined (sum in quadrature)		**10.2**
Repeatability (calibration of B10K10) (c)	A	0.9
Repeatability (calibration of B10K12)	A	0.9

Table 7: Summary of the NIMT uncertainty budget.

In Table 7, the uncertainty (a) associated with the reference standard Rs is the total uncertainty for the measurement of Rs in terms of the QHR. The contribution from the uncertainty on the temperature of oil (b) is a function of the relative temperature coefficient of Rs, namely $\alpha(R\mathrm{s}) = 1.5 \times 10^{-6}$/°C. The uncertainty associated with the repeatability is calculated from the standard deviation of the mean of the series of measurements.

NIMT After corrections	Relative difference from nominal value / 10^{-6}	Relative standard uncertainties		
		Repeatability u_1 / 10^{-8}	Systematic u_2 / 10^{-8}	Corrections u_3 / 10^{-8}
B10K10	0.796	0.9	10.2	2.0
B10K12	1.184	0.9	10.2	2.0

Table 8: Summary of the NIMT results, after corrections for temperature and pressure.

For a single standard, the NIMT uncertainty $u_{\mathrm{NIMT},\,i}$ is obtained from: $u^2_{NIMT,\,i} = u^2_{1,\,i} + u^2_{2,\,i} + u^2_{3,\,i}$
Unlike the $u_{1,\,i}$, the $u_{2,\,i}$ and $u_{3,\,i}$ are assumed to be correlated.

Using expression (2), when the mean (for two standards) of the NIMT-BIPM relative difference is calculated, the NIMT contribution to the uncertainty is:

$$u^2_{\mathrm{NIMT}} = \sum_{i=1}^{2} \frac{u^2_{1,\,i}}{2^2} + u_2^2 + u_3^2 \qquad (5)$$

Using the values shown in Table 8 the relative standard uncertainty u_{NIMT} is

$$u_{\mathrm{NIMT}} = 10.4 \times 10^{-8}.$$

4 Comparison NIMT – BIPM

4.1 Results

The differences between the values assigned by the NIMT at the NIMT, R_{NIMT}, and those assigned by the BIPM at the BIPM, R_{BIPM}, to each of the two travelling standards during the period of the comparison are shown in Table 9.

NIMT - BIPM	
Standard #	$(R_{\mathrm{NIMT}} - R_{\mathrm{BIPM}})$ / (10 kΩ) / 10^{-6}
B10K10	+ 0.629
B10K12	+ 0.682
mean	**+ 0.656**

Table 9: Differences between the values assigned by the NIMT (R_{NIMT}) and by the BIPM (R_{BIPM}) to the two travelling standards.

The mean difference between the NIMT and the BIPM calibrations is:

$$\mathbf{(R_{NIMT} - R_{BIPM}) / (10\ k\Omega) = +0.66 \times 10^{-6}}$$

The relative combined standard uncertainty of the comparison, u_{C}, is:

$$u_{\mathrm{C}}^2 = u_{\mathrm{BIPM}}^2 + u_{\mathrm{NIMT}}^2 + u_{\mathrm{T}}^2$$

where u_{BIPM} = 1.5 x 10^{-8},
u_{NIMT} = 10.4 x 10^{-8},
u_{T} = 0.22 x 10^{-8}
as calculated in sections 2 and 3: $\mathbf{u_{C} = 0.105 \times 10^{-6}}$

The final result of the comparison is presented as the degree of equivalence D between the NIMT and the BIPM for values assigned to 10 kΩ resistance standards, and its expanded relative uncertainty (expansion factor k = 2, corresponding to a confidence level of 95 %) , U_{C}

$$\mathbf{D = [(R_{NIMT} - R_{BIPM}) / 10\ k\Omega] = +0.66 \times 10^{-6}}$$

$$\mathbf{U_{C} = 0.21 \times 10^{-6}}$$

The difference between the NIMT and the BIPM calibration results is significantly larger than the expanded uncertainty.

4.2 Follow-up investigations at the NIMT

After publication of Draft A in May 2009, NIMT started a series of checks and investigations to determine the origin of this large discrepancy. The preliminary conclusions were sent to the BIPM in November 2009.
It was found that the main source of error is most likely related to a failure of the temperature monitoring system of their oil-bath. A difference of 0.2 °C to 0.3 °C was observed between the monitored and the actual temperature. The travelling standards are measured by comparison with a NIMT 1000 Ω reference standard, and this ratio changes by about 0.3 x 10^{-6} to 0.5 x 10^{-6} for such a temperature difference.
Other problems have been experienced with the power supply of the magnet in the NIMT QHR system. It was noticed that the applied current does not correspond exactly to the produced magnetic field.

4.3 Conclusions

The difference between the NIMT and the BIPM calibration results is significantly larger than the expanded uncertainty.
However, this exercise allowed previously undetected sources of errors to be discovered in the NIMT facility. A new bilateral comparison can be organized as soon as these problems are fixed.

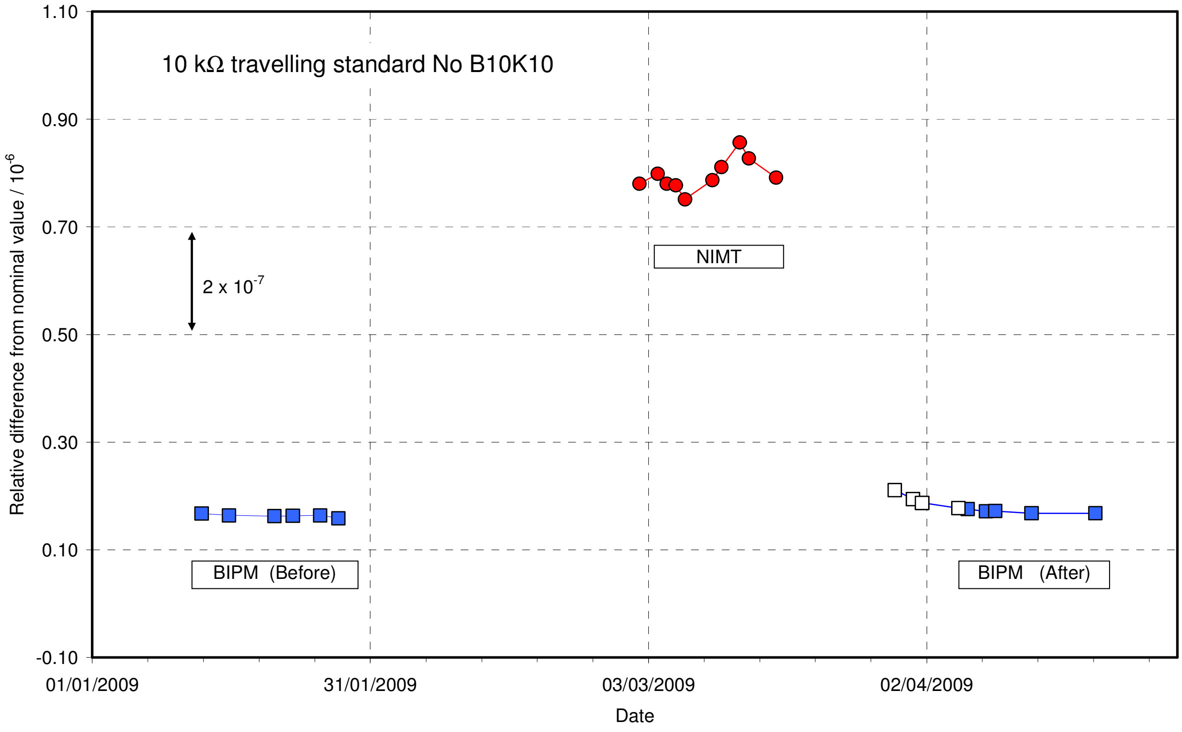

Figure 1: Calibrations at the BIPM (squares) and at the NIMT (circles) of the travelling standard ref. B10K10, expressed as the relative deviation from the nominal 10 kΩ value. The white squares correspond to transient values not used in the calculation.

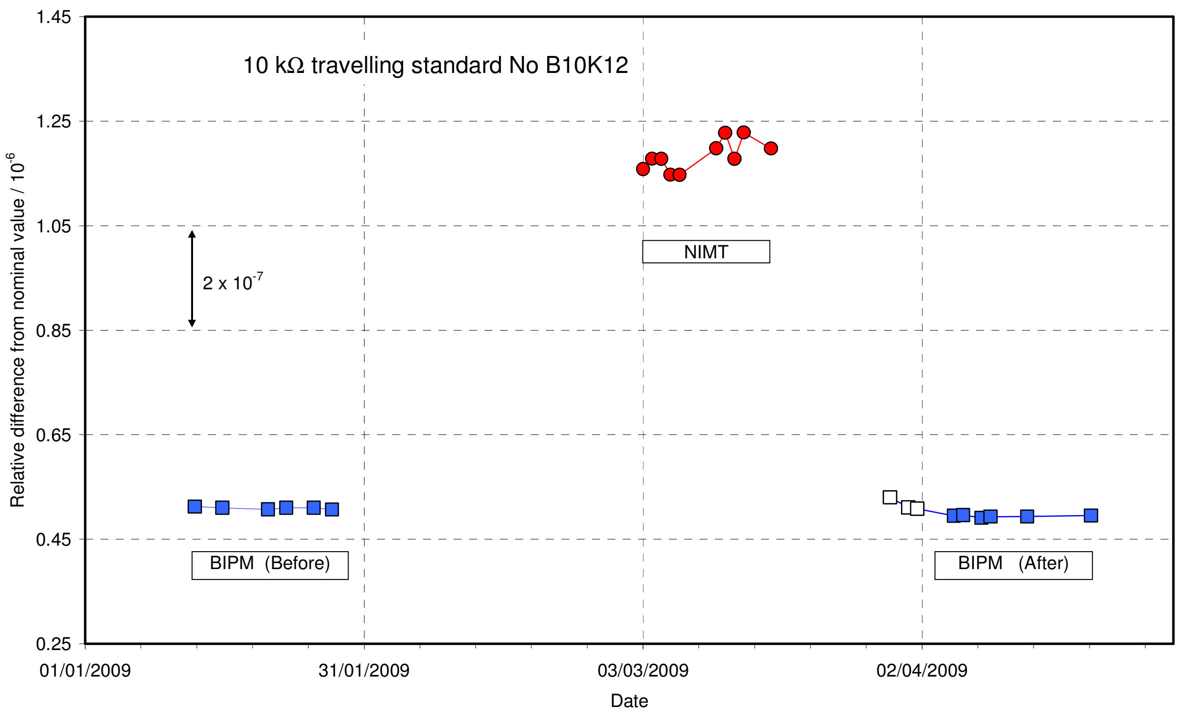

Figure 2: Calibrations at the BIPM (squares) and at the NIMT (circles) of the travelling standard ref. B10K12, expressed as the relative deviation from the nominal 10 kΩ value. The white squares correspond to transient values not used in the calculation.