BUREAU INTERNATIONAL
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Bilateral comparison of 10 kΩ standards
(ongoing BIPM key comparison BIPM.EM-K13.b)
between the NML (Ireland) 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 NML-Ireland (NMLI) in the period September 2008 to December 2008.

Two 10 kΩ BIPM travelling standards (ESI, SR104 type) were calibrated first at the BIPM, then at the NMLI and again at the BIPM after their return. The measurement periods are referred to as:

'Before' measurements at the BIPM: September-October 2008
NMLI measurements: October-November 2008
'After' measurements at the BIPM: November-December 2008

The BIPM calibrations are corrected to the reference temperature 23.000 °C and the reference pressure 1013.25 hPa.
According to the protocol, the NMLI 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 NMLI.

The calibration reports provided by the NMLI 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', 'NMLI' and 'After': see Figures 1 and 2). In particular, the two standards exhibited a significant increase of their resistance after their return to the BIPM, and a subsequent decrease during about one week, down to a stable value. The values corresponding to this transient period (white diamonds on Figure 1 and Figure 2) have not been used in the calculation. The measurement period 'After' starts on the 5 December 2008 (blue diamonds on the Figures).

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 NMLI and the BIPM calibrations of a given standard R_i can be written as:

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

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

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

This expression can also be written as:

$$\Delta_{\mathrm{NMLI-BIPM}} = \frac{1}{2}\sum_{i=1}^{2}R_{\mathrm{NMLI},\,i} - \frac{1}{2}\sum_{i=1}^{2}R_{\mathrm{BIPM},\,i} \tag{2}$$

which is the difference of the means.

The reference standards of the two participants are closely correlated, as the NMLI takes its traceability from the BIPM. The effect of this correlation is reduced by the length of time since the last comparison of NMLI's standards with those of the BIPM, in April 2006.

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 September 2008, 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 (within 0.04 °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}	St. d. mean u_{1B} / 10^{-9}	AFTER / 10^{-6}	St. d. mean u_{1A} / 10^{-9}
B10K08	+ 0.563	2	+ 0.558	2
B10K09	− 0.372	1	− 0.307	2
Mean value of 'Before' and 'After'				
Standard #	**mean** / 10^{-6}	Exp. Std. dev. u_1 / 10^{-9}	Systematic u_2 / 10^{-9}	
B10K08	+ 0.561	1	15	
B10K09	− 0.340	1	15	

Table 2: Summary of the BIPM calibrations. The dispersion is estimated by the standard deviations, 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:

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

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

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

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

Using expression (2), when the mean (for two standards) of the NMLI-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 Uncertainty associated with the transfer

u_{d} is the uncertainty associated with any uncompensated drift or step changes in the values of the travelling standards, as observed by the BIPM.
The final resistance value attributed by the BIPM is the arithmetic mean of the 'Before' and 'After' measurements.
As we have no clear knowledge about the behaviour of the standards during the period between the BIPM 'Before' and 'After' measurements, the value assigned by the BIPM to each standard, on the mean date of the comparison, is taken to lie, with equal probability, in an interval of width $d = \left|\left(R_{After} - R_{Before}\right)\right|$ centred on the mean value.

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

Another source of uncertainty associated with the transfer can be the difference in the operating currents used by the two laboratories, influencing the resistance of the standards through their power coefficients. In the present case, the nominal operating current is 0.5 mA at the NMLI and 0.1 mA at the BIPM. Based on estimations for previous comparisons of the same type, the value of the relative standard uncertainty u_{P} associated with possible power effects is estimated to be $u_{\mathrm{P}} = 10 \times 10^{-9}$.

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

The $u_{\mathrm{P},\,i}$ are assumed to be correlated, unlike $u_{\mathrm{d},\,i}$.
Following the same reasoning as in expression (3), the uncertainty u_{T} associated with the transfer (for the mean of two standards) is: $u^2_{\mathrm{T}} = \sum_{i=1}^{2} \frac{u^2_{\mathrm{d},\,i}}{2^2} + u^2_{\mathrm{P}}$

	Transfer	
Standard #	Drift u_{d} / 10^{-9}	Power u_{P} / 10^{-9}
B10K08	1	10
B10K09	19	10
Combined	9	10
Total u_{T}	**14**	

Table 3: Uncertainty associated with the transfer.

Using the values of Table 3, the relative standard uncertainty u_T is:

$$u_T = 14 \times 10^{-9}$$

3 Measurements at the NMLI

3.1 Method of calibration:

The travelling standards were allowed to stabilise in air at an ambient temperature of (23 ±1) °C. The resistance of each standard was measured by comparison with the NMLI 10 kΩ reference standard. The comparison of the travelling standards with the reference group was carried out using a substitution measuring technique. A resistance bridge, based on a binary divider (MIL Model 6000A) was used as transfer standard.
The temperature of the travelling standard was measured by means of a digital platinum resistance thermometer, whose sensor was placed in the resistor's thermometer well.

3.2 Operating conditions:

Operating current: 0.5 mA dc.
Atmospheric pressure range: 990 hPa – 1019 hPa.

3.3 NMLI results:

The standards were measured 12 times in the period 31 October – 24 November 2008.
The results are summarized in Table 4.
The standard uncertainty u_1 refers to the experimental standard deviation of the mean and u_2 to the other sources of uncertainty listed in Table 7.

Serial No. of standard	Mean date of measurement	Resistance value (Rx /10 kΩ) – 1 /10^{-6}	Mean temperature / °C	Mean barometric pressure / hPa	Experimental std.dev. mean u_1 / 10^{-6}	Standard uncertainty u_2 / 10^{-6}
B10K08	8 Nov 2008	+ 0.589	22.41	1009	0.009	0.30
B10K09	8 Nov 2008	– 0.243	22.27	1009	0.014	0.30

Table 4: Summary of the NMLI calibrations.

The NMLI results are corrected to the reference temperature and the reference pressure using the coefficients shown in Table 5, according to:

$$R(23) = R(T) - \alpha_{23} \cdot (T - 23) - \beta \cdot (T - 23)^2$$

where $R(T)$ is the resistance measured at the temperature T.

The corrections, calculated by the BIPM, are shown in Table 6.

	Relative temperature coefficients		Relative pressure coefficients.
Standard #	Alpha $_{23}$ / (10^{-6}/K)	Beta / (10^{-6}/K²)	/ (10^{-9}/hPa)
B10K08	– 0.010	– 0.023	– 0.162
B10K09	– 0.040	– 0.022	– 0.164

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

Reference temperature = 23.000°C Reference pressure = 1013.25 hPa		
	Relative corrections applied to the NMLI results	
Standard #	For temperature	For pressure
B10K08	$+ 0.004 \times 10^{-6}$	$- 0.001 \times 10^{-6}$
B10K09	$- 0.015 \times 10^{-6}$	$- 0.001 \times 10^{-6}$

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

Taking into account the uncertainty reported by the NMLI for temperature corrections normally applied by this laboratory, and the estimated uncertainty for pressure corrections, the uncertainty u_3 associated with the temperature and pressure corrections is estimated to be $u_3 = 0.01 \times 10^{-6}$.

Source of uncertainty	Relative standard uncertainty / 10^{-6}
NMLI 10 kΩ reference standard Rs	0.200
Ratio measurement Rx to Rs	0.200
Correction for leakage effects	0.100
Combined: u_2 =	0.300

Table 7: Summary of the NMLI uncertainty budget associated with the comparison of a standard Rx with the NMLI reference Rs. The uncertainty associated with temperature and pressure corrections is not included here.

NMLI After corrections	Relative difference from nominal value / 10^{-6}	Relative standard uncertainties		
		Dispersion u_1 / 10^{-6}	Systematic u_2 / 10^{-6}	Corrections u_3 / 10^{-6}
B10K08	+ 0.592	0.009	0.30	0.01
B10K09	− 0.259	0.014	0.30	0.01

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

For a single standard, the NMLI uncertainty $u_{\mathrm{NMLI},\,i}$ is obtained from: $u^2_{NMLI,\,i} = u^2_{1,\,i} + u^2_{2,\,i} + u^2_{3,\,i}$

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

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

$$u^2_{\mathrm{NMLI}} = \sum_{i=1}^{2} \frac{u^2_{1,\,i}}{2^2} + u_2^2 + u_3^2 \qquad (4)$$

Using the values shown in Table 8, it is clear that the contributions from the dispersion (u_1) and the corrections (u_3) are almost negligible compared to u_2.

The relative standard uncertainty u_{NMLI} is

$$u_{\mathrm{NMLI}} = 0.30 \times 10^{-6}.$$

4 Comparison NMLI – BIPM

4.1 Data reduction using the arithmetic mean:

The differences between the values assigned by the NMLI at the NMLI, R_{NMLI}, 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.

Standard #	$\Delta_i = (R_{\text{NMLI}} - R_{\text{BIPM}})$ / (10 kΩ) / 10^{-6}
B10K08	+ 0.031
B10K09	+ 0.081
mean	+ 0.056

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

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

$$(R_{\text{NMLI}} - R_{\text{BIPM}}) / (10\ \text{k}\Omega) = +0.056 \times 10^{-6}$$

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

$$u_{\text{C}}^2 = u_{\text{BIPM}}^2 + u_{\text{NMLI}}^2 + u_{\text{T}}^2 \qquad (5)$$

where $u_{\text{BIPM}} = 0.015 \times 10^{-6}$,
$u_{\text{NMLI}} = 0.30 \times 10^{-6}$,
$u_{\text{T}} = 0.014 \times 10^{-6}$
as calculated in Sections 2 and 3: $u_{\text{C}} = 0.30 \times 10^{-6}$

4.2 Data reduction using a weighted mean:

In Section 4.1, the two differences Δ_1 and Δ_2 have the same weight in the calculation of the mean, and their uncertainties (slightly different due to different transfer uncertainties) are combined according to the classical expressions (3) and (5).

In another approach, more confidence can be given to a result obtained from a standard showing a better stability (with a lower transfer uncertainty), so that the weights attributed to Δ_1 and Δ_2 would be different.

More generally, Δ_1 and Δ_2 are strongly (but not completely) correlated quantities.
Their mean can be calculated using the generalized weighted mean of correlated quantities (1), described below:

$\overline{X} = \sigma_{\bar{x}}^2 \left(W^T C^{-1} X \right)$ is the weighted mean,

where:

$\sigma_{\bar{x}}^2 = \left(W^T C^{-1} W \right)^{-1}$ is the associated variance,

$W = \begin{pmatrix} 1 \\ \vdots \\ 1 \end{pmatrix}$ is the design matrix

C is the covariance matrix

$$X = \begin{pmatrix} x_1 \\ : \\ x_n \end{pmatrix} \text{ the series of data}$$

In order to calculate the covariance matrix, the contributions to the uncertainties associated with Δ_1 and Δ_2, that is $u(\Delta_1)$ and $u(\Delta_2)$ respectively, are grouped in Table 10.

Those marked with $^{(**)}$ are assumed to be fully correlated between $u(\Delta_1)$ and $u(\Delta_2)$.
The uncertainties associated with the dispersion of the measurements in each laboratory (Type A evaluation) and with transport (Type B evaluation of a random effect) are assumed to be uncorrelated between $u(\Delta_1)$ and $u(\Delta_2)$.

Uncertainties Type A, B		Δ_1 / 10^{-6}	Δ_2 / 10^{-6}
u_1 BIPM	A	0.001	0.001
u_2 BIPM	B $^{(**)}$	0.015	0.015
u_1 NMLI	A	0.010	0.013
u_2 NMLI	B $^{(**)}$	0.300	0.300
u_3 (corrections)	B $^{(**)}$	0.010	0.010
u_p (power effects)	B $^{(**)}$	0.010	0.010
u_{Tranfer}	B	0.001	0.019
Combined (quadratic sum) :			
All components:	$u(\Delta_i)$	0.30088	0.30159
Correlated $^{(**)}$ components	$u_{\text{Corr.}}(\Delta_i)$	0.30071	0.30071

Table 10: Contributions to the uncertainties associated with Δ_1 and Δ_2
(numerical values are taken from Tables 2, 3 and 8).

The covariance matrix is then written as:

$$C = \begin{pmatrix} u(\Delta_1)^2 & u_{Corr.}^2 \\ u_{Corr.}^2 & u(\Delta_2)^2 \end{pmatrix} ; \qquad \text{and } W = \begin{pmatrix} 1 \\ 1 \end{pmatrix} ; \qquad X = \begin{pmatrix} \Delta_1 \\ \Delta_2 \end{pmatrix} ;$$

with $\Delta_1 = 0.031 \times 10^{-6}$
$\Delta_2 = 0.081 \times 10^{-6}$

Using the values shown in Table 10, the weighted mean and the associated standard deviation are:

$\overline{X} = 0.040 \times 10^{-6}$
$\sigma = 0.30 \times 10^{-6}$

that is:

$(R_{\text{NMLI}} - R_{\text{BIPM}}) / (1\ \Omega) = + 0.039 \times 10^{-6}$

$u_C = 0.30 \times 10^{-6}$

As expected, more weight was given to Δ_1 which is associated with a smaller transfer uncertainty.

After discussion between the BIPM and the NMLI, this calculation method was chosen to express the final result.

The final result of the comparison is presented as the degree of equivalence D between the NMLI 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

$$\boldsymbol{D = [(R_{\mathrm{NMLI}} - R_{\mathrm{BIPM}}) / 10\ \mathrm{k\Omega}] = +\,0.039 \times 10^{-6}}$$

$$\boldsymbol{U_{\mathrm{C}} = 0.60 \times 10^{-6}}$$

The NMLI and the BIPM calibrations are in good agreement, with a difference smaller than the expanded uncertainty.

References:

[(1)] The generalized weighted mean of correlated quantities, M.G. Cox et al., Metrologia **43** (2006), S268-S275.

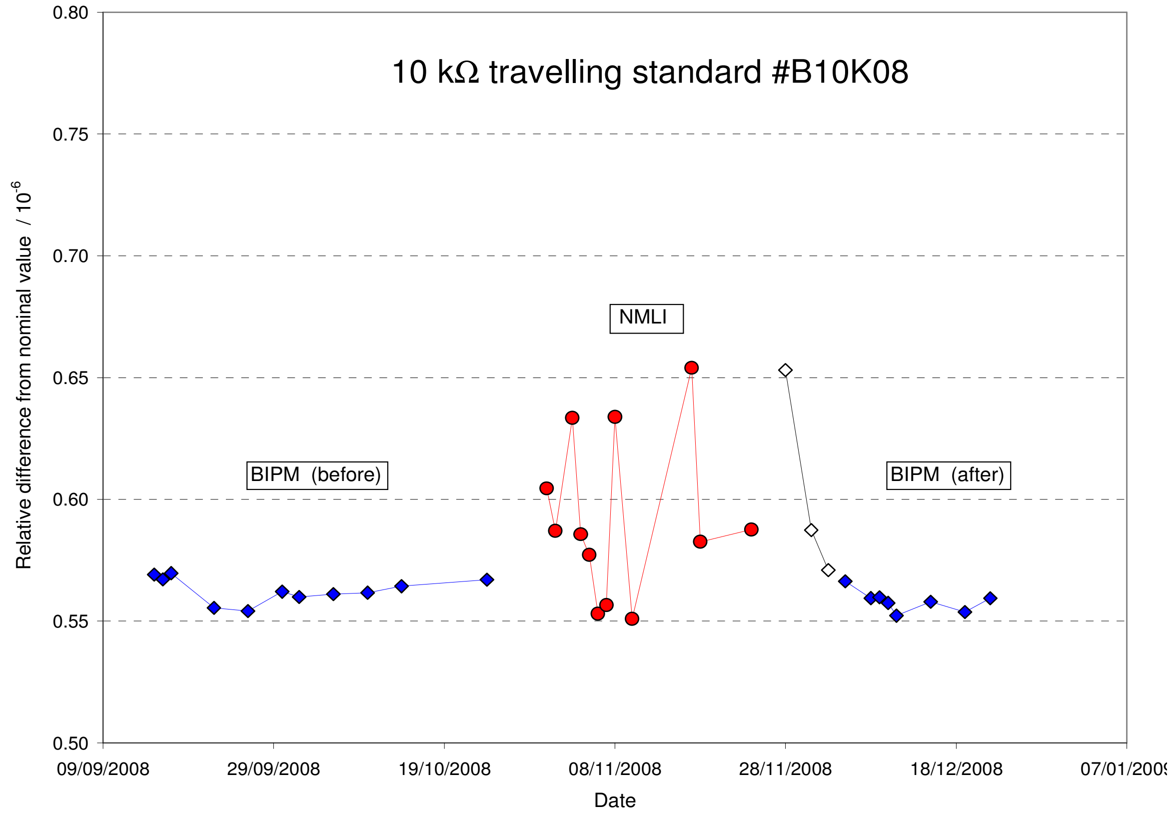

Figure 1: Calibrations at the BIPM (diamonds) and at the NMLI (circles) of the travelling standard ref. B10K08, expressed as the relative deviation from the nominal 10 kΩ value. The white diamonds correspond to transient values not used in the calculation.

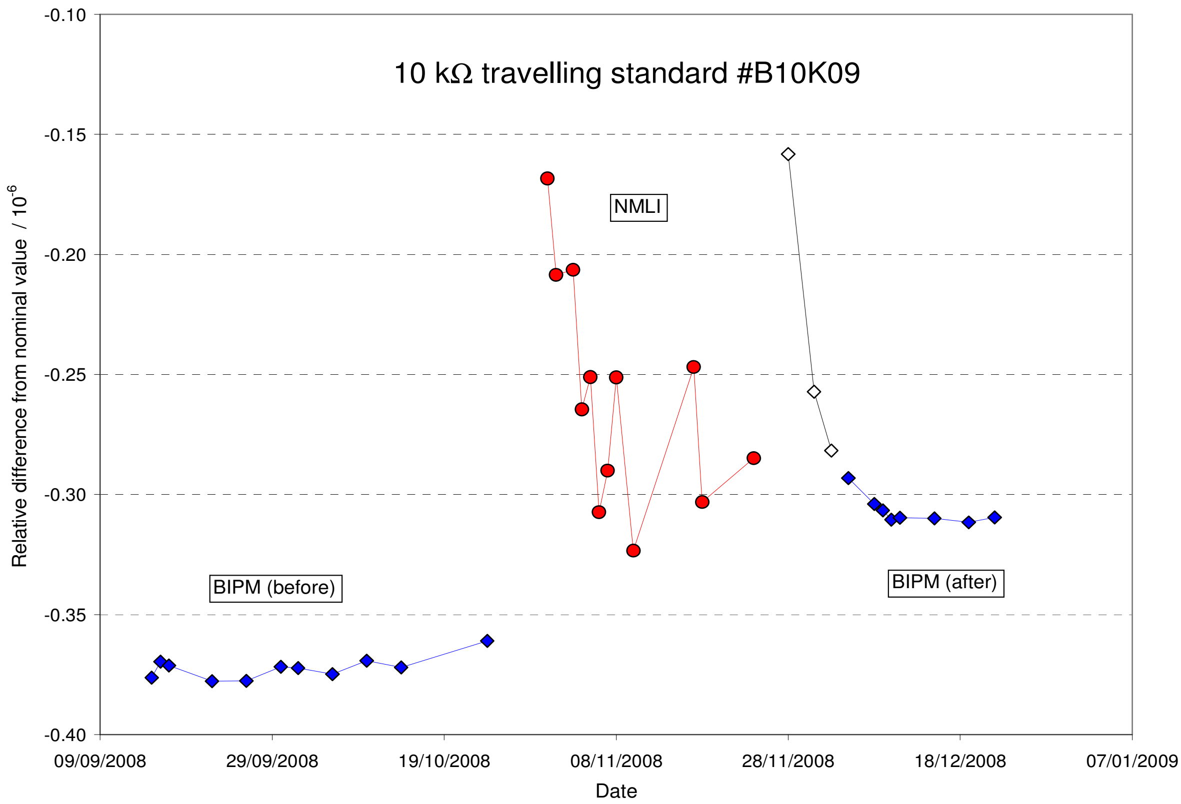

Figure 2: Calibrations at the BIPM (diamonds) and at the NMLI (circles) of the travelling standard ref. B10K09, expressed as the relative deviation from the nominal 10 kΩ value. The white diamonds correspond to transient values not used in the calculation.