BUREAU INTERNATIONAL DES POIDS ET MESURES

Bilateral Comparison of 10 kΩ standards (ongoing BIPM key comparison BIPM.EM-K13.b) between the CMI (Czech Republic) and the BIPM

May 2008

R. Goebel^{*}, P. Chrobok^{**}, N. Fletcher^{*} and M. Stock^{*}.

* Bureau International des Poids et Mesures, Sèvres, France. ** Czech Metrology Institute, Prague, Czech Republic.



1 Introduction

A comparison of values assigned to $10 \text{ k}\Omega$ resistance standards was carried out between the BIPM and the CMI (Czech Republic) in the period January 2008 to April 2008.

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

'Before' measurements at the BIPM: January 2008 CMI measurements: February - March 2008 'After' measurements at the BIPM: March 2008 - April 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 CMI 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 CMI.

The calibration reports provided by the CMI 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', 'CMI' and 'After': see Figures 1 and 2). Moreover, the two standards exhibited a significant increase 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 diamonds on Figure 1 and Figure 2) have not been used in the calculation. The measurement period 'After' starts on the 28 March 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 CMI and the BIPM calibrations of a given standard R_i can be written as:

$$\Delta_i = R_{\text{CMI},i} - R_{\text{BIPM},i}$$

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

$$\Delta_{\text{CMI-BIPM}} = \frac{1}{2} \sum_{i=1}^{2} (R_{\text{CMI},i} - R_{\text{BIPM},i})$$
(1)

This expression can also be written as:

$$\Delta_{\rm CMI-BIPM} = \frac{1}{2} \sum_{i=1}^{2} R_{\rm CMI, i} - \frac{1}{2} \sum_{i=1}^{2} R_{\rm BIPM, i}$$
(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 k Ω 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 k Ω reference was calibrated against the QHR in January 2008, during the first part of the comparison.

The 10 k Ω travelling standards were kept in a temperature controlled air bath at a temperature which is close (less than 0.03 °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.

Source of uncertainty	relative standard uncertainty / 10 ⁻⁹
Imperfect realization of $R_{\rm H}(2)$	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 $10 \text{ k}\Omega$ 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 <i>u</i> ₂	15 × 10 ⁻⁹

The BIPM measurements are summarized in Table 2 and the uncertainty budget in Table 1.

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

BIPM	BIPM Relative difference from nominal 10 kΩ value					
Standard #	BEFORE / 10 ⁻⁶	Std. u_{1B}	dev. / 10 ⁻⁹	AFTEI / 10 ⁻⁶	R	Std. dev. $u_{1A} / 10^{-9}$
B10K08	0.50244		7	0.5493	4	7
B10K11	0.63841		5	0.6462	9	8
	Mean value of 'Before' and 'After'					
Standard #	mean / 10 ⁻⁶		Exp. u_1	Std. dev. / 10 ⁻⁹	ŝ	Systematic $u_2 / 10^{-9}$
B10K08	0.5259			5		15
B10K11	0.6423		5			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.

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

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

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

 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 $u_{\text{BIPM}, i}$ is obtained from: $u_{\text{BIPM}, i}^2 = u_{1, i}^2 + u_{2, i}^2$ The $u_{2, i}$ are assumed to be correlated, unlike $u_{1, i}$.

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

$$u_{\rm BIPM}^2 = \sum_{i=1}^2 \frac{u_{1,i}^2}{2^2} + u_2^2$$
(3)

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

$$u_{\rm BIPM} = 15.4 \times 10^{-9}$$

2.2 Uncertainty associated with the transfer

 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 28 March 2008 (blue diamonds 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: $d = |(R_{After} - R_{Before})|$

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,

 $u_{\rm d} = \frac{d}{2} \cdot \frac{1}{\sqrt{3}}$

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 0.3 mA at the CMI and 0.1 mA at the BIPM. Based on estimations for previous comparisons of the same type, the value of the relative standard uncertainty $u_{\rm P}$ associated with possible power effects is estimated to be $u_{\rm P} = 3 \times 10^{-9}$.

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

The $u_{P,i}$ are assumed to be correlated, unlike $u_{d,i}$.

Following the same reasoning as in expression (3), the uncertainty u_T associated with the transfer (for the mean of two standards) is:

$$u_{\rm T}^2 = \sum_{i=1}^2 \frac{u_{\rm d,i}^2}{2^2} + u_{\rm P}^2$$

	Transfer		
Standard #	Drift $u_{\rm d} / 10^{-9}$	Power $u_{\rm P} / 10^{-9}$	
B10K08	14	3	
B10K11	2	3	
Combined	7.1	3	
Total <i>u</i> _T	7.7		

Table 3: Uncertainty associated with the drift and
the power coefficient of the standards.

Using the values of Table 3, the relative standard uncertainty $u_{\rm T}$ is:

 $u_{\rm T} = 7.7 \times 10^{-9}$

3 Measurements at the CMI

3.1 Method of calibration:

The resistance standards placed in a temperature controlled laboratory at 23 °C were measured by indirect comparison with a 100 Ω reference standard using a Quantum Hall Resistance Bridge Model MI 6010 Q, repeatedly, in a four-terminal configuration. The 100 Ω reference standard is itself known in terms of the recommended value of the von Klitzing constant, $R_{K-90} = 25\,812.807\,\Omega$.

3.2 Operating conditions:

Operating current: 0.3 mA dc. Atmospheric pressure range: 964 hPa to 992 hPa.

3.3 <u>CMI results</u>:

The standards were measured 12 times in the period 22 February - 17 March 2008. The results are summarized in Table 4.

Serial No. of standard:	Resistance value (Ω)	Repeatability / 10 ⁻⁹	Mean temperature / °C	Mean atmospheric pressure / hPa
B10K08	10 000. 005 11	7	23.02	980
B10K11	10 000. 006 17	7	22.99	980

Table 4: Summary of the CMI calibrations.

The CMI results are corrected to the reference temperature and the reference pressure using the coefficients shown in Table 5. The corrections, calculated by the BIPM, are shown in Table 6.

	Relative temperatu	Relative pressure coefficients.	
Standard #	Alpha ₂₃ / ($10^{-6}/K$) Beta / ($10^{-6}/K^{2}$)		/ (10 ⁻⁹ /hPa)
B10K08	- 0.010	- 0.023	- 0.162
B10K11	-0.070	- 0.027	- 0.281

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 CMI results		
Standard #	For temperature For pressure	
B10K08 $+ 0.3 \times 10^{-9}$ $- 5.3 \times 10^{-9}$		
B10K11 -1.0×10^{-9} -9.3×10^{-9}		

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

The uncertainties on temperature and pressure measurements at the CMI are 0.05 °C and 2 hPa respectively.

Taking into account the differences from the reference temperature and reference pressure, the uncertainties u_{Temp} and u_{Press} associated with the temperature and pressure corrections applied by the BIPM are estimated to be $u_{\text{Temp}} = 6 \times 10^{-9}$ and $u_{\text{Press}} = 7 \times 10^{-9}$, leading to a combined uncertainty $u_3 = 9 \times 10^{-9}$.

	Relative standard
Source of uncertainty	$/ 10^{-9}$
Reference standard R _s uncertainty	15.0
Drift of R _s	11.6
Ratio 1000 / 100	11.6
Linearity of ratio 1000 / 100	5.8
Ratio 10 000 / 1000	11.6
Linearity of ratio 10 000 / 1000	5.8
Air temperature on R _s	2.9
Power dissipation on R _s	5.8
Atmospheric pressure on R _s	5.8
Leakage currents	5.8
Connection error	5.8
Combined (sum in quadrature) <i>u</i> ₂	29
Repeatability u_1	7

Table 7: Summary of the CMI uncertainty budget

CMI	Relative difference from	stan	Relative dard uncertain	ties
After corrections	nominal value / 10 ⁻⁶	Dispersion $u_1 / 10^{-9}$	Systematic $u_2 / 10^{-9}$	Corrections $u_3 / 10^{-9}$
B10K08	0.5060	7	29	9
B10K11	0.6073	7	29	9

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

For a single standard, the CMI uncertainty $u_{\text{CMI}, i}$ is obtained from: $u_{\text{CMI}, i}^2 = u_{1, i}^2 + u_{2, i}^2 + u_{3, i}^2$ The $u_{2, i}$ and $u_{3, i}$ are assumed to be correlated, unlike the $u_{1, i}$.

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

$$u_{\rm CMI}^2 = \sum_{i=1}^2 \frac{u_{1,i}^2}{2^2} + u_2^2 + u_3^2$$
(5)

Using the values shown in Table 8 the relative standard uncertainty $u_{\rm CMI}$ is

$$u_{\rm CMI} = 30.8 \times 10^{-9}$$
.

4 <u>Comparison CMI – BIPM</u>

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

CMI - BIPM		
Standard #	$(R_{ m CMI} - R_{ m BIPM}) / (10 \ m k\Omega) / 10^{-6}$	
B10K08	- 0.0199	
B10K11	- 0.0351	
mean	- 0.028	

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

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

 $(R_{\rm CMI} - R_{\rm BIPM}) / (10 \text{ k}\Omega) = -0.028 \times 10^{-6}$

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

$$u_{\rm C}^2 = u_{\rm BIPM}^2 + u_{\rm CMI}^2 + u_{\rm T}^2$$

where $u_{\text{BIPM}} = 15.4 \times 10^{-9}$, $u_{\text{CMI}} = 30.8 \times 10^{-9}$, $u_{\text{T}} = 7.7 \times 10^{-9}$ as calculated in sections 2 and 3: $u_{\text{C}} = 0.035 \times 10^{-6}$

The final result of the comparison is presented as the degree of equivalence *D* between the CMI and the BIPM for values assigned to 10 k Ω resistance standards, and its expanded relative uncertainty (expansion factor *k* = 2, corresponding to a confidence level of 95 %), *U*_C

$$D = [(R_{\rm CMI} - R_{\rm BIPM}) / 10 \text{ k}\Omega] = -0.028 \times 10^{-6}$$
$$U_{\rm C} = 0.070 \times 10^{-6}$$

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



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



Figure 2: Calibrations at the BIPM (diamonds) and at the CMI (circles) of the travelling standard ref. B10K11, expressed as the relative deviation from the nominal 10 k Ω value. The white diamonds correspond to transient values not used in the calculation.