

# BUREAU INTERNATIONAL DES POIDS ET MESURES

## On-site comparison of Quantum Hall Effect resistance standards of the CMI and the BIPM

◆◆ Ongoing key comparison BIPM.EM-K12 ◆◆

*Report on the April 2017 on-site comparison*

*Final Report, October 2017*

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### 1. Introduction

The ongoing on-site comparison BIPM.EM-K12 is part of the BIPM programme implemented to verify the international coherence of the primary resistance standards. It allows National Metrology Institutes (NMIs) to validate their implementations of the Quantum Hall Effect (QHE) for dc resistance traceability by comparison to the reference maintained at the BIPM.

In this comparison, the realization of the ohm from the QHE-based standard of the NMIs at 100  $\Omega$  is compared with that realized by the BIPM from its own transportable quantum Hall resistance standard. This comparison is completed by scaling measurements from 100  $\Omega$  to 1  $\Omega$  and 10 k $\Omega$ .

The comparison programme BIPM.EM-K12 started in 1993. A first series of five comparisons were carried out from this date until 1999. After a suspension period, the comparison was resumed in 2013. Since then one comparison has been successfully completed, one had to be abandoned due to a problem with the participant's measurement system and one had to be postponed upon the participant's request. Available results may be consulted on the Key Comparison Data Base (KCDB) webpage [1].

In April 2017 a new BIPM.EM-K12 comparison was carried out at the Czech Metrology Institute (CMI). This report presents the measurement results obtained during this exercise.

### 2. Principle of the comparison measurements

The ohm can be reproduced from the QHE routinely with an accuracy of the order of 1 part in  $10^9$  or better. The present comparison is performed on-site in order to eliminate the limitation of transporting transfer resistance standards between the BIPM and the participating institute, which would otherwise result in an increase by at least a factor of 10 of the comparison uncertainty.

To this end, the BIPM has developed a complete transportable system that can be operated at the participant's facilities to reproduce the ohm from a QHE reference at 100  $\Omega$  and scale this value to 1  $\Omega$  and 10 k $\Omega$  (meaning that not only the QHE systems are covered in this comparison but also the scaling devices).

Practically, the comparison comprises three stages schematized in figure 1:

- (i) The calibration of a 100  $\Omega$  standard resistor in terms of the QHE based standard of each of the institutes (CMI and BIPM).

The conventional value  $R_{K-90}$  is used to define the quantum Hall resistance value. The relative difference in the calibrated values of the standard resistor of nominal value  $R=100 \Omega$  is expressed as  $(R_{CMI} - R_{BIPM}) / R_{BIPM}$  where  $R_{BIPM}$  and  $R_{CMI}$  are the values attributed by the BIPM and CMI respectively.

- (ii) The scaling from 100  $\Omega$  to 10 k $\Omega$ , through the measurement of the ratio  $R_{10k\Omega} / R_{100\Omega}$  of the resistance of two standards of nominal value 10 k $\Omega$  and 100  $\Omega$ . The relative difference in the measurement of this ratio, hereinafter referred to as  $K1$ , is expressed as  $(K1_{CMI} - K1_{BIPM}) / K1_{BIPM}$  where  $K1_{BIPM}$  and  $K1_{CMI}$  are the values attributed by the BIPM and the CMI respectively.

- (iii) The scaling from  $100\ \Omega$  to  $1\ \Omega$ , through the measurement of the ratio  $R_{100\ \Omega} / R_{1\ \Omega}$  of the resistance of two standards of nominal value  $100\ \Omega$  and  $1\ \Omega$ . The relative difference in the measurement of this ratio, hereinafter referred to as  $K2$ , is expressed as  $(K2_{\text{CMI}} - K2_{\text{BIPM}}) / K2_{\text{BIPM}}$  where  $K2_{\text{BIPM}}$  and  $K2_{\text{CMI}}$  are the values attributed by the BIPM and the CMI respectively.

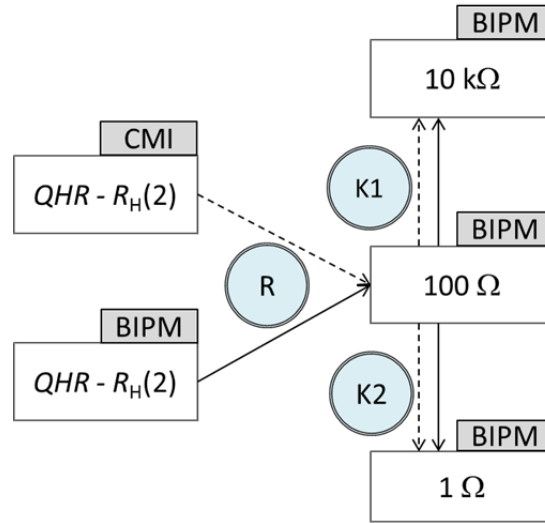


Figure 1: Schematic of the onsite comparison carried out at the CMI in April 2017. Rectangles represent the resistances to be compared and circles correspond to the resistance  $R$  or the ratios  $K1$  and  $K2$  to be measured. Solid and dashed arrows stand for the measurements with the 1 Hz-bridge of the BIPM or with the CCC bridge of the CMI, respectively.

The resistance value of each of the standard resistors used in this comparison is defined as its five-terminal dc-resistance value<sup>1</sup>. This means that it corresponds to the dc voltage to current ratio once any thermal emf across the resistor, particularly those induced by Peltier effect, has reached a stable value. As will see later on in this report the estimation of the dc-resistance value of a resistor, or a ratio of resistors, may be vitiated by a significant measurement error especially for the  $1\ \Omega$  standard. This issue has been the subject of several papers [2, 3, 4] in which an extended description of the observed phenomena is provided.

### 3. The BIPM measurement system and the transfer standards

#### 3.1. Implementation of the QHE

A complete transportable QHE reference [5] has been developed at the BIPM for the purpose of the BIPM.EM-K12 on-site comparison programme. It is composed of a compact liquid helium cryostat equipped with an 11 tesla magnet and a sample space that can be cooled to 1.3 K with the included vacuum pump (the superconducting magnet has an additional support at the bottom of the dewar to allow safe transport).

The separate sample probe can support two TO-8 mounted quantum Hall devices simultaneously (side by side within the magnet), with guarded wiring for eight terminals on each device. The BIPM uses GaAs heterostructure devices fabricated in the LEP 1990 EUROMET batch [6]. They give an  $i=2$  plateau centered around 10.5 T which is well quantized for currents of at least  $100\ \mu\text{A}$  at 1.5 K. The cryostat and the QHE

<sup>1</sup> Ratio of the voltage drop between the high and low potential terminals to the current flowing in the low current terminal, with the case - fifth terminal - maintained at the same potential as the low potential terminal

devices are suitable for a realization of the ohm ( $\Omega$ -90) meeting all the requirements of the CCEM guidelines [7] for a relative standard uncertainty down to  $1 \times 10^{-9}$ .

A transportable resistance bridge is used with the QHE cryostat for the measurement of the different resistance ratios being the subject of the comparison. It is based on a room-temperature low-frequency current comparator (LFCC) operated at 1 Hz (sinusoidal signal), meaning that all resistance or ratio measurements are carried out at 1 Hz by the BIPM during the comparison. That way to proceed is preferable to the transport of the BIPM CCC bridge on-site since the 1Hz-bridge is a more rugged instrument, simple to operate, and much less sensitive to electromagnetic interference and temperature variations. Furthermore, it provides resolution and reproducibility that are comparable to those achievable with the BIPM CCC.

The 1 Hz-bridge is equipped with two separate LFCCs of ratio 129:1 and 100:1, having turns 2065/16 and 1500:15. The construction and performances of these devices are detailed in [8, 9].

### 3.2. Transfer standards

Three transfer resistance standards of value 1  $\Omega$ , 100  $\Omega$  and 10 k $\Omega$  are used during the comparison. The values assigned by the BIPM and the CMI to the 100  $\Omega$  resistor in terms of  $R_{K-90}$  and to the two ratios 100  $\Omega$  / 1  $\Omega$  and 10 k $\Omega$  / 100  $\Omega$  are the measurands being compared in this comparison.

The transfer standards were provided by the BIPM. The 1  $\Omega$  standard was of CSIRO-type (s/n: S-64202) and the 100  $\Omega$  and the 10 k $\Omega$  standards were Tegam resistors of type SR102 (s/n: A2030405) and SR104 (s/n: K204039730104), respectively. All three resistors were fitted in individual temperature-controlled enclosures held at 25°C. The temperature-regulation system might be powered either from the mains or from external batteries.

For each of these standards, the difference between resistance values measured at 1 Hz and at 'dc' is small but not negligible. These differences were determined at the BIPM prior to the comparison and checked after. The 'dc' value was measured with the BIPM cryogenic current comparator (CCC) whilst the 1 Hz value with the transportable 1Hz-bridge subsequently used onsite during the comparison. The differences are applied as corrections to the measurements performed at 1 Hz meaning that the 1Hz-bridge is used as a transfer instrument referenced to the BIPM CCC.

The frequency corrections (1 Hz-'dc') are reported in Table 1 for each of the three transfer standards. The main possible error sources contributing to these corrections are the quantum Hall resistance (QHR), the 1 Hz-bridge and the transfer standard itself. Nevertheless, at 1 Hz, the frequency dependence of the QHR is negligible compared to the comparison uncertainty [10], and the characterization of the bridge evidenced that its error at 1 Hz is below 1 part in  $10^9$ . Consequently, the frequency dependence observed is mainly due to the resistance standards themselves.

Resistance or Resistance ratio	1 Hz - 'dc' Correction / $10^{-6}$	Standard Uncertainty / $10^{-9}$
100 $\Omega$	0.0101	1.0
10 000 $\Omega$ / 100 $\Omega$	-0.0098	1.0
100 $\Omega$ / 1 $\Omega$	-0.0245	2.0

Table 1: Value of the 1 Hz to 'dc' corrections (in relative) applied to the BIPM measurements carried out at 1 Hz. These values are specific to the standards used in the present comparison.

For the sake of completeness, it must be noticed that the 'dc' resistance value (or ratio) measured with the BIPM CCC-bridge results from a current signal passing through the resistors having polarity reversals with

a waiting time between polarity inversions, cf. figure 2. The polarity reversal frequency is of the order of 3 mHz (340 s cycle period) and the measurements are sampled only during 100 s before the change of polarity.

Previous characterization measurements of the  $R_H(2) / 100 \Omega$  and  $10 \text{ k}\Omega / 100 \Omega$  ratios have shown that if the polarity reversal frequency is kept below 0.1 Hz, then any effects of settling or ac behaviour remain of the order of 1 part in  $10^9$  or less. Regarding the  $100 \Omega / 1 \Omega$  ratio this is most often not the case due to unavoidable Peltier effects in the  $1 \Omega$  standard.

Consequently, in order to ensure the best possible comparability of the measurements performed by the BIPM and the participating institute, the measuring system of the latter should be ideally configured to match the reference polarity reversal cycle of the BIPM CCC. As this is generally not possible, a correction must be applied on the participating institute's measurements based on additional characterization of the influence of the polarity reversal rate on the actual measured resistance ratio.

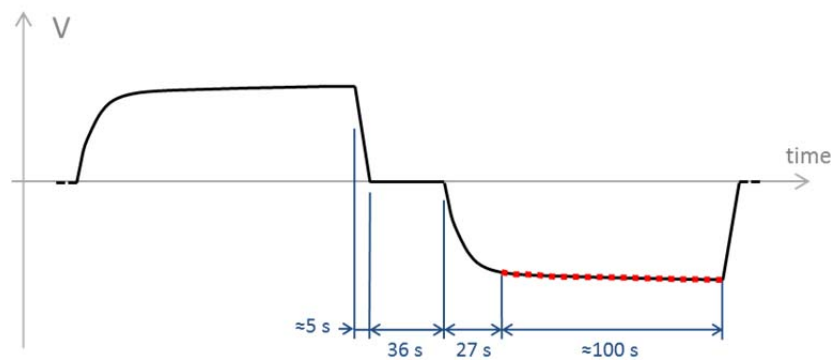


Figure 2: Schematic representation of the reference signal current with polarity reversals used in the BIPM CCC-bridge. The reversal cycle comprises a waiting time of about 36 s at zero current. The red dotted line corresponds to the sampling time period

### 3.3. Uncertainty budget

Table 2 summarizes the BIPM standard uncertainties for the measurement of the 'dc' value of the  $100 \Omega$  standard in terms of the recommended value of the von Klitzing constant  $R_{K-90}$ , as well as the measurement uncertainties for both the  $100 \Omega / 1 \Omega$  and  $10 \text{ k}\Omega / 100 \Omega$  ratios.

Parameters	Ratio	Relative standard uncertainties / $10^{-9}$		
		$R_H(2)/100 \Omega$	$10 \text{ k}\Omega/100 \Omega$	$100 \Omega/1 \Omega$
Reference CCC bridge				
Imperfect CCC winding ratio		1.0	1.0	1.0
Resistive divider calibration		0.5	0.5	0.5
Leakage resistances		0.2	0.2	-
Noise rectification in CCC		1.0	1.0	1.0
Imperfect realization of the QHR		1.0	-	-
Correction of the 1 Hz-to 'dc' difference		1.0	1.0	2.0
<b>Combined type B standard uncertainty, <math>u_B</math>=</b>		<b>2.1</b>	<b>1.8</b>	<b>2.5</b>

Table 2: Contributions to the combined type B uncertainty ( $k=1$ ) for the measurement of the three mentioned resistance ratios at the BIPM.

#### 4. The CMI measurement system

The CMI measurement system is similar to the one used during preliminary BIPM.EM-K13.a and 13.b bilateral comparisons performed with the BIPM in 2015 [11]. It is an improved version of [12] but with additional modifications brought by CMI. In particular, a new cryogenic insert (VSM12) allows the QHR device to be operated now at a temperature between 2.3 K and 4.2 K. Also, a modification of cabling between the QHR system, the measurement bridge CCC-1 (from Magnicon GmbH, www.magnicon.com) and the resistance standards under calibration led to improved properties of the CMI measurement system.

Estimation of uncertainty sources of the measurement bridge CCC-1 was based mainly on calibration of its compensation unit [13], tests of winding ratio errors, grounding currents effects and channel symmetry. The bridge configurations used during the comparison together with the associated sources of uncertainty of measurements are summarized in Table 3.

<i>Parameters</i>	<i>Ratio</i>	$R_H(2)/100\ \Omega$	10 k $\Omega$ /100 $\Omega$	100 $\Omega$ /1 $\Omega$
Number of turns $N_1/N_2$		2065/16	1600/16	200/2
Number of turns $N_A$		1	1	1
DUT voltage (V)		0.3	0.5	0.05
Compensation ratio		0.0258484	0.001293	-0.0001402
<i>Type-B contributions / 10<sup>-9</sup></i>				
CCC winding ratio		1.0	1.0	1.0
CCC electronics and SQUID		0.6	0.6	0.6
Compensation ratio k		0.4	<0.1	<0.1
Bridge voltage measurement		<0.1	<0.1	<0.1
Measurement of the DUT voltage		<0.1	<0.1	<0.1
Leakage resistance		0.3	0.3	0.1
Imperfect realization of $R_H(2)$		1.2	-	-
<b>Combined type B standard uncertainty, <math>u_B</math>=</b>		<b>1.3</b>	<b>1.2</b>	<b>1.2</b>

Table 3: Bridge configurations and type-B contributions ( $k=1$ ) to the uncertainty budget for the CMI measurements of the mentioned resistance ratios.

The shape of the standard current reversal cycle of the measurement bridge of CMI is schematized in figure 3. The full cycle length is about 24 s and the reversal time between positive and negative current plateaus is about 0.4 s. The bridge voltage is evaluated from measured voltage data points laying between the 4th and 12th second of each current plateau time.

Details of the standard reversal cycle timing, hereinafter referred to as configuration “A<sub>24</sub>”, together with other experimental timing configurations also used, are stated in Table 4. Explanation of the timing parameters is reported in Figure 3.

Configuration B<sub>340</sub> was chosen as an equivalent of BIPM’s ‘dc’ CCC-bridge cycle during the on-site measurement campaign and configurations C<sub>10</sub>, D<sub>6</sub> and E<sub>5</sub> were used for the study of possible effects on the measurement of  $R_H(2) / 100\ \Omega$ , K1 and K2 ratios.

Property/Configuration	A <sub>24</sub>	B <sub>340</sub>	C <sub>10</sub>	D <sub>6</sub>	E <sub>5</sub>
$t_{FC}$ – full cycle time (s)	24	340	10	6	5.2
$t_{HC}$ – half cycle time (s)	12	170	5	3	2.6
$t_R$ – ramp time (s)	0.4	10	0.4	0.4	0.4
$t_W$ – waiting time before sampling (s)	3.8	53	1.6	1	0.8
$t_S$ – sample time (s)	8	107	3	1.6	1.4

Table 4: Timing details of the CCC reversal cycles used by CMI during the comparison. Stated values are approximated and can slightly vary due to measurement system timing, up to 1 % for longest cycles and up to 15 % for shortest cycles.

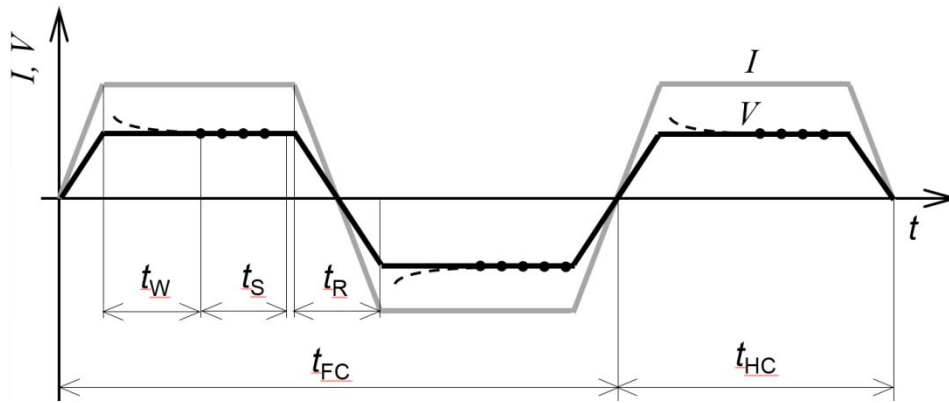


Figure 3: Timing of the measurement cycle of CMI bridge, where  $I$  and  $V$  curves corresponds to ideal shapes of measurement current and respective unbalanced bridge voltage. Dashed line shows slow transients which are apparent in real measurement of  $V$ . Dots correspond to sampled data used for statistics ( $t_{FC}$  – full cycle time,  $t_{HC}$  – half cycle time,  $t_R$  – ramp time,  $t_W$  – waiting time before sampling,  $t_S$  – sample time).

## 5. Measurement of the 100 $\Omega$ transfer standard in terms of $R_H(2)$

### 5.1. BIPM measurements

#### 5.1.1. Preliminary tests

The quantum Hall sample used during the present comparison was operated on the  $i=2$  plateau at a temperature of 1.3 K and with a rms current of 40  $\mu\text{A}$ . The magnetic flux density corresponding to the middle of the plateau was determined by recording the longitudinal voltage  $V_{xx}$  versus flux density and was found to be 10.5 T. The two-terminal Hall resistance of the four-terminal-pairs device was checked before and after each series of measurements, showing that the contact resistance was smaller than a few ohms (and in any case not larger than 5  $\Omega$  - measurements limited by the resolution of the DVM used). The absence of significant longitudinal dissipation along both sides of the device was tested as described in [7] section 6.2, by combining the measurements obtained from four different configurations of the voltage contacts (two opposite pairs in the center and at the end of the sample, and two diagonal configurations). The absence of dissipation was demonstrated within  $5 \times 10^{-10}$  in relative terms with a standard deviation of the same order. Subsequent series of measurements were taken from the central pair of contacts only.

### 5.1.2. BIPM results

As mentioned above, the quantum Hall sample was used with a rms current of 40  $\mu\text{A}$ . The current in the 100  $\Omega$  transfer standard was then 5.2 mA, corresponding to a Joule heating dissipation of 2.7 mW.

After a preliminary set of measurements on 25 April 2017, five measurements of the 100  $\Omega$  standard were interleaved with four measurements by CMI on 26 April 2017. The 1 Hz-measured and dc-corrected values of the 100  $\Omega$  standard are reported in Table 5. They are expressed as the relative difference from the 100  $\Omega$  nominal value:  $(R_{\text{BIPM}} / 100 \Omega) - 1$ .

Each measurement reported in the table below is the mean value of eight individual measurements corresponding to a total integration time of about 40 minutes.

Time	$(R_{\text{BIPM}}/100 \Omega) - 1 \quad /10^{-6}$		Dispersion $/10^{-9}$
	1 Hz measurements	'dc' corrected (1 Hz-'dc' correction)	
10:38	-0.634 74	-0.624 66	1.1
12:09	-0.635 25	-0.625 17	1.8
13:52	-0.635 97	-0.625 89	2.1
15:26	-0.635 01	-0.624 93	1.1
17:11	-0.635 32	-0.625 24	1.5
<b>Mean value =</b>		<b>-0.625 18</b>	
<b>Standard deviation, <math>u_A</math> =</b>		<b><math>0.21 \times 10^{-9}</math></b>	

Table 5 : BIPM measurements of the 100  $\Omega$  standard in terms of  $R_{\text{H}}(2)$ , on 26 April 2017. Results are expressed as the relative difference from the nominal 100  $\Omega$  value.

BIPM result :  $R_{\text{BIPM}} = 100 \times (1 - 0.625 18 \times 10^{-6}) \Omega$

Relative standard uncertainty :  $u_{\text{BIPM}} = 2.1 \times 10^{-9}$

where  $u_{\text{BIPM}}$  is calculated as the quadratic sum of  $u_A = 0.21 \times 10^{-9}$  and, from Table 2,  $u_B = 2.1 \times 10^{-9}$ .

## 5.2. CMI measurements of $R_{\text{H}}(2)/100 \Omega$

### 5.2.1. Preliminary tests

The GaAs-based quantum Hall device QHR P579-101 (from PTB) with Sn-ball contacts was used for this comparison. It was operated on the  $i = 2$  plateau. The magnetic flux density corresponding to the middle of the plateau was determined by recording the longitudinal voltage as a function of magnetic flux at current level  $\pm 100 \mu\text{A}$ , and was found to be 9.52 T. Possible temperature, current and geometric dependencies of QHR device quantization [7] were evaluated by means of  $R_{\text{xy}}$  measurements at temperatures between 2.3 K and 4.2 K, current levels of 23  $\mu\text{A}$  and 39  $\mu\text{A}$ , and by combining measurements taken from opposite pairs and diagonal pairs of voltage contacts. The measurements of  $R_{\text{xy}}$  were performed against a 100  $\Omega$  reference resistance standard with known properties belonging to the CMI.

Standard measurement current flowing through the QHR during the comparison was 23  $\mu\text{A}$  (3 mA in the 100  $\Omega$  standard) and the device was operated most of the time at a temperature of 4.2 K. Only for evaluation of the current coefficient of BIPM's 100  $\Omega$  transfer standard a current of 39  $\mu\text{A}$  was also applied. Estimated total uncertainty of imperfect realization of  $R_{\text{H}}(2)$  is reported in Table 3.



### 5.2.2. Influence of the measurement current

Significantly different current levels were used by CMI and BIPM during the measurement of the 100 Ω transfer standard, implying different Joule heating dissipation in this resistor. The influence of the applied current on its value was then evaluated.

The relative change of resistance of the 100 Ω standard measured against QHR, while the current is varied from 3 mA to 5 mA, was estimated to (QHR maintained at about 2.5 K):

$$R(5 \text{ mA})/R(3 \text{ mA}) - 1 = (-1.19 \pm 0.32) \times 10^{-9} \quad (k=1)$$

### 5.2.3. Possible influence of the measurement cycle duration

As mentioned earlier, measurement results can be influenced by reversal cycle timing. This effect was investigated in [3, 4, 8] and pointed out during a previous on-site comparison [2].

The effect of reversal cycle timing on the value of the 100 Ω standard was then estimated for different reversal cycle configurations. Results are presented in Table 6 in the form of relative differences to the standard reversal cycle A<sub>24</sub>. As can be seen, there is a small but still significant difference that has to be taken into account, especially for the configuration B<sub>340</sub>/A<sub>24</sub>, for which the difference corresponds to the correction to be applied to the standard A<sub>24</sub> measurement to obtain the corrected 'dc' equivalent measurement for the reference cycle time of 340 s.

Possible effects of resistance value fluctuation during the measurements were eliminated by means of interleaved or repeated measurements in the different cycle configurations. In the case of repeated measurements, a weighted mean of the measured values was taken as a result of the evaluation.

Combinations x, y of reversal cycles	δ : rel. diff. of R(x)/R(y)-1 /10 <sup>-9</sup>	Std. uncertainty of δ (k=1) /10 <sup>-9</sup>
B <sub>340</sub> /A <sub>24</sub>	<b>+0.92</b>	0.35
A <sub>24</sub> /C <sub>10</sub>	+0.7	0.70
A <sub>24</sub> /D <sub>6</sub>	+1.51	0.29

Table 6: Effect of varying reversal cycle configuration on the 100 Ω standard measurements in terms of R<sub>H</sub>(2). Measurement carried out with CMI measuring system. Notations D<sub>6</sub>, C<sub>10</sub>, A<sub>24</sub> and B<sub>340</sub> refer to Table 4.

### 5.2.4. CMI results for standard measurement cycle duration

Four measurements (interleaved with the five BIPM measurements of table 5) were taken on 26 April 2017, each consisting of 80 standard measurement cycles. The overall measurement time corresponding to those 80 cycles was about 33 minutes. The current through the 100 Ω standard was 3 mA.

Table 7 presents the measurement results obtained under these conditions as well as their value after correction: correction for the different measurement currents, 3 mA and 5 mA, and correction for the different cycle times, from 24 s to the reference equivalent 'dc' cycle time (340 s).

Time	$(R_{\text{CMI}}/100 \Omega)-1 \quad /10^{-6}$		Dispersion / $10^{-9}$
	Standard cycle time (24 s) & 3 mA	'dc' & 'current' corrected (340 s) & 5 mA	
11:10	-0.625 12	-0.625 39	0.20
12:39	-0.625 56	-0.625 83	0.23
14:20	-0.625 63	-0.625 90	0.23
16:01	-0.625 53	-0.625 80	0.22
<b>Mean value =</b>		<b>-0.625 73</b>	
<b>Standard deviation, <math>u_A</math> =</b>		<b><math>0.12 \times 10^{-9}</math></b>	

Table 7: CMI measurements of the 100  $\Omega$  standard in terms of  $R_{\text{H}}(2)$  on 26 April 2017. Results are expressed as the relative difference from the nominal 100  $\Omega$  value.

CMI result:  $R_{\text{CMI}} = 100 \times (1 - 0.625 73 \times 10^{-6}) \Omega$

Relative standard uncertainty:  $u_{\text{CMI}} = 1.4 \times 10^{-9}$

where  $u_{\text{CMI}}$  is calculated as the quadratic sum of:  $u_A = 0.12 \times 10^{-9}$ ,  $u_B = 1.3 \times 10^{-9}$  (from Table 3),  $u_i = 0.32 \times 10^{-9}$  the standard uncertainty on the current correction, and  $u_{\text{CT}} = 0.35 \times 10^{-9}$  the standard uncertainty on cycle time correction.

### 5.3. 100 $\Omega$ measurements comparison

Figure 4 presents the corrected interleaved measurements from CMI and BIPM on 26 April 2017 (from data in tables 5 and 7). Error bars correspond to the dispersion observed for each measurement. The dispersion of the individual BIPM measurements in the actual experimental conditions at CMI was about twice as usual. However, the dispersion on the series of five measurements had a negligible effect on final combined comparison uncertainty.

We then suggest from figure 4 that within the limit of the dispersion of the results, the stability of the 100  $\Omega$  transfer resistor is such that any uncertainty contribution from possible instabilities can be estimated as negligible.

The difference between CMI and BIPM can then be calculated as the difference of the means of the series of measurements carried out by both institutes (mean values reported in tables 5 and 7):

Relative difference CMI-BIPM:  $(R_{\text{CMI}} - R_{\text{BIPM}}) / R_{\text{BIPM}} = -0.6 \times 10^{-9}$

Relative combined standard uncertainty:  $u_{\text{comp}} = 2.5 \times 10^{-9}$

where  $u_{\text{comp}}$  is calculated as the quadratic sum of  $u_{\text{BIPM}} = 2.1 \times 10^{-9}$  and  $u_{\text{CMI}} = 1.4 \times 10^{-9}$ .

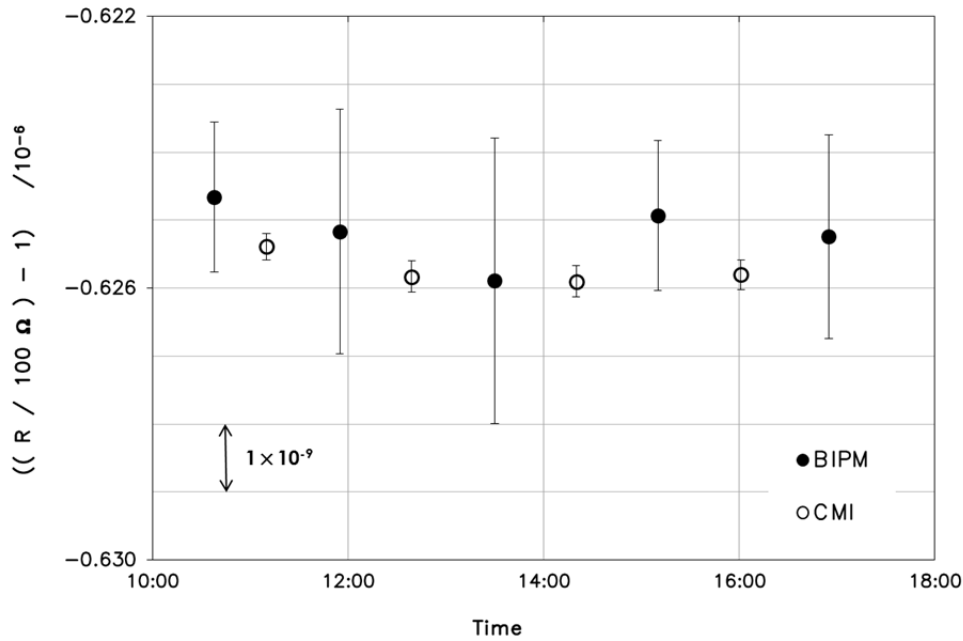


Figure 4: CMI (white circles) and BIPM (black dots) corrected measurements of the 100  $\Omega$  resistance  $R$  in terms of  $R_H(2)$  on 26 April 2017. The uncertainty bars correspond to the dispersion observed during each measurement.

## 6. Measurement of the (10 000 $\Omega$ / 100 $\Omega$ ) ratio $K1$

### 6.1. BIPM measurements of $K1$

For the measurement of the  $K1$  ratio the 129:1 LFCC equipping the BIPM's 1 Hz-bridge for  $R_H(2) / 100 \Omega$  ratio measurement was replaced by a 100:1 current comparator. The rms current in the 10 000  $\Omega$  standard was 50  $\mu\text{A}$  corresponding to 5 mA in the 100  $\Omega$  standard. The two standards were connected alternately to the BIPM and CMI bridges. Six BIPM measurements were interleaved with five CMI measurements.

As in the previous section, each measurement result reported in Table 8 corresponds to the mean value of 8 individual measurements corresponding to a total integration time of about 40 minutes. The associated dispersion corresponds to the standard deviation of the mean of the 8 measurements.

Time	$(K1_{\text{BIPM}}/100)-1 / 10^{-6}$		Dispersion $/10^{-9}$
	1 Hz measurements	'dc' corrected (1 Hz-'dc' correction)	
9 :41	0.815 00	0.805 25	0.84
11 :27	0.813 50	0.803 75	0.60
13 :18	0.813 27	0.803 52	0.56
15 :14	0.815 25	0.805 50	0.84
17 :03	0.815 13	0.805 38	1.00
19 :12	0.815 37	0.805 62	1.00
<b>Mean value =</b>		<b>0.804 84</b>	
<b>Standard deviation, <math>u_A</math> =</b>		<b><math>0.38 \times 10^{-9}</math></b>	

Table 8: BIPM measurements of the (10 000  $\Omega$  / 100  $\Omega$ ) ratio  $K1$  on 27 April 2017. Results are expressed as the relative difference from the nominal ratio value 100.

BIPM result:  $K1_{\text{BIPM}} = 100 \times (1 + 0.804\,84 \times 10^{-6})$

Relative standard uncertainty:  $u_{\text{BIPM}} = 1.8 \times 10^{-9}$

where  $u_{\text{BIPM}}$  is calculated as the quadratic sum of  $u_A = 0.38 \times 10^{-9}$  and, from Table 2,  $u_B = 1.8 \times 10^{-9}$ .

## 6.2. CMI measurements of K1

### 6.2.1. CMI results for standard cycle duration

Five measurements (interleaved with six BIPM measurements) were taken on 27 April 2017, each consisting of 100 measurement cycles. Results for a standard cycle time are given in Table 9. The overall measurement time of 100 cycles was about 41 minutes. The measurement current through 10 000  $\Omega$  standard was 50  $\mu\text{A}$ , as for the BIPM.

Time	$(K1_{\text{CMI}}/100)-1$ / $10^{-6}$		Dispersion / $10^{-9}$
	Standard cycle time (24s)	'dc' corrected	
10:17	0.808 29	0.806 19	0.70
12:02	0.808 63	0.806 53	0.58
14:01	0.808 26	0.806 16	0.77
15:47	0.807 06	0.804 96	0.65
17:45	0.808 04	0.805 94	0.47
<b>Mean value =</b>		<b>0.805 96</b>	
<b>Standard deviation, <math>u_A</math> =</b>		<b><math>0.27 \times 10^{-9}</math></b>	

Table 9: CMI measurements of the (10 000  $\Omega$  / 100  $\Omega$ ) ratio K1 on 27 April 2017. Results are expressed as the relative difference from the nominal ratio value 100.

### 6.2.2. Possible influence of the cycle duration

As for  $R_{\text{H}}(2) / 100 \Omega$  ratio, the effect of reversal cycle timing on the measurement of K1 ratio was estimated. It was found that the relative difference between K1 measurements when the cycle time is varied from the 24 s standard duration to the reference 340 s duration is equal to  $-2.1 \times 10^{-9}$ , see table 10. This difference is significant and was used as a correction of K1 measurements to obtain the 'dc' equivalent K1 value. Possible effects of resistance value fluctuation during the measurements were eliminated by means of interleaved measurements in A<sub>24</sub> and B<sub>340</sub> configurations.

Configuration x, y of cycle timing	$\delta$ : rel. diff. of $K1(x)/K1(y)-1$ / $10^{-9}$	Std. uncertainty of $\delta$ ( $k=1$ ) / $10^{-9}$
B <sub>340</sub> /A <sub>24</sub>	-2.1	0.35

Table 10: Effect of varying reversal cycle configuration on K1 ratio measurement. Measurements are carried out with CMI measuring system. Notations A<sub>24</sub> and B<sub>340</sub> refer to Table 4.

### 6.2.3. CMI results for 'dc' equivalent cycle duration (340 s)

The equivalent 'dc' corrected measurements are reported in table 9 together with the results for standard reversal cycle timing.

CMI are then finally:  $K1_{\text{CMI}} = 100 \times (1 + 0.805\,96 \times 10^{-6}) \Omega$

Relative standard uncertainty:  $u_{\text{CMI}} = 1.3 \times 10^{-9}$

where  $u_{\text{CMI}}$  is calculated as the quadratic sum of  $u_A = 0.27 \times 10^{-9}$ ,  $u_B = 1.2 \times 10^{-9}$  (from Table 3), and  $u_{\text{CT}} = 0.35 \times 10^{-9}$  the standard uncertainty on cycle time correction.

### 6.3. Comparison of K1 measurements

Figure 5 presents the corrected interleaved measurements from CMI and BIPM on 27 April 2017 (data from tables 8 and 9). Error bars correspond to the dispersion observed for each measurement.

It can be noticed that differences are more important for the first series of measurements. However the largest difference still remains less than the interval covered by CMI and BIPM uncertainty bars (of the order of  $2 \times 10^{-9}$  and  $1.5 \times 10^{-9}$  for BIPM and CMI, respectively - not shown on the graph). We then suggest that no significant instabilities of the standards can be evidenced and therefore no additional uncertainty component was included in the final result.

The difference between CMI and BIPM can then be calculated as the difference of the means of the series of measurements carried out by both institutes and reported in tables 8 and 9:

Relative difference CMI-BIPM:  $(K1_{\text{CMI}} - K1_{\text{BIPM}}) / K1_{\text{BIPM}} = +1.1 \times 10^{-9}$

Relative combined standard uncertainty:  $u_{\text{comp}} = 2.2 \times 10^{-9}$

where  $u_{\text{comp}}$  is calculated as the quadratic sum of  $u_{\text{BIPM}} = 1.8 \times 10^{-9}$  and  $u_{\text{CMI}} = 1.3 \times 10^{-9}$ .

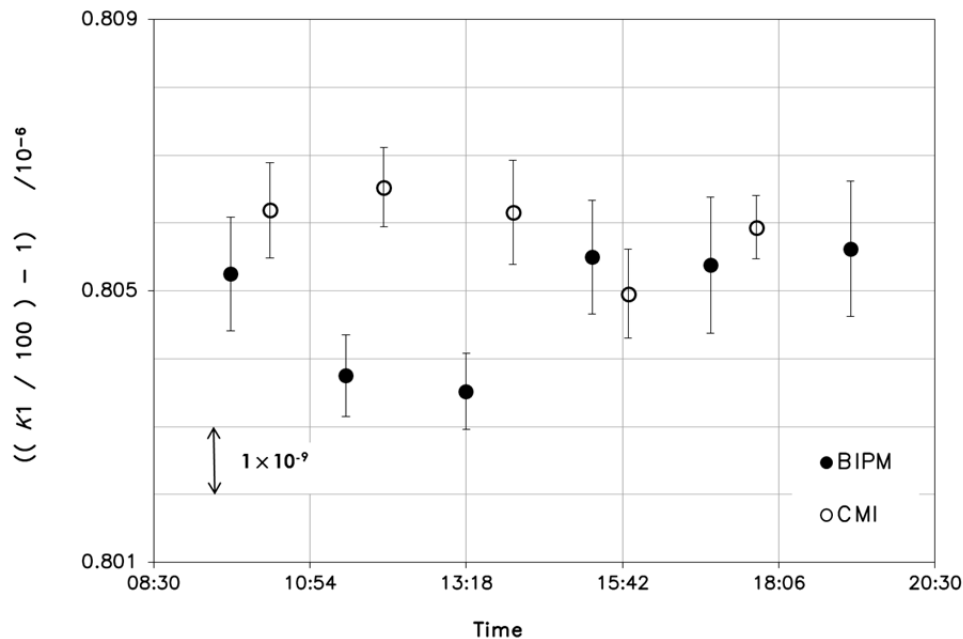


Figure 5: CMI (white circles) and BIPM (black dots) corrected measurements of the ratio K1 (10 000 Ω / 100 Ω) on 27 April 2017. The uncertainty bars correspond to the dispersion observed during each measurement

## 7. Measurement of the (100 Ω / 1 Ω) ratio *K2*

### 7.1. Influence of the cycle time duration

The influence of the Peltier effect on the measured value of 1 Ω resistors of the type used in this comparison (CSIRO-type) has been observed and described several times in previous works [2, 3, 14]. This effect is dependent on the cycle time duration and on the delay after reversing the current in dc measurements and may be as high as several parts in 10<sup>8</sup> (relative difference of the 1 Ω value between short - but >10 s - and long cycle times). As a consequence, the measured value of the 100 Ω / 1 Ω ratio is also impacted to the same extent as Peltier effect remains negligible in the 100 Ω resistor (typically 1 to few parts in 10<sup>9</sup>, depending on the resistor - as found in section 5.2.3).

This has been clearly evidenced and quantified during a previous BIPM.EM-K12 comparison between BIPM and PTB [2]. In particular, further investigations carried out during this comparison and subsequently led to the conclusion that the conditions of 'dc resistance measurements' of ratio *K2* were not sufficiently well-defined in regard to the resolution of the two measurement systems in comparison (the equivalent 'dc resistance measurement' conditions were the same as in the present comparison). On the contrary, results obtained have evidenced a better comparability of *K2* ratio measurements of both institutes for the shortest cycle times investigated in the comparison (i.e. 1 Hz for BIPM and about 0.2 Hz for PTB).

Although a different 1 Ω transfer standard (but of the same construction type) was used during the present comparison, the measuring system operated by CMI was very similar to that used during the above mentioned one between BIPM and PTB and we expected to arrive at a comparable conclusion. For this reason, measurements of ratio *K2* were carried out once again by CMI not only for the standard cycle time but also for additional ones ranging from 5.2 s to 340 s. The other conditions of measurement were the same as those used for the measurement of *K1* ratio but with a nominal current of 50 mA in the 1 Ω standard.

The influence of the reversal cycle timing on the measurement of the *K2* ratio is reported in table 11. Figure 6 presents the variation of *K2* versus cycle time deduced from the *K2* ratio value measured for the standard cycle time - *A*<sub>24</sub> - and the relative differences of table 11. The measurement of the *K2* ratio using the CMI standard cycle time is given in table 14 (section 7.4).

The BIPM measurement of *K2* at 1 Hz and the 'dc' corrected *K2* value for the reference 340 s cycle time are also reported in figure 6 (see 7.4 for conditions of measurement).

Configuration x, y of cycle timing	$\delta$ : rel. diff. of $K2(x)/K2(y)-1$ /10 <sup>-9</sup>	Std. uncertainty of $\delta$ ( $k=1$ ) /10 <sup>-9</sup>
B <sub>340</sub> /A <sub>24</sub>	-34.8	0.7
A <sub>24</sub> /C <sub>10</sub>	-6.0	0.9
A <sub>24</sub> /E <sub>5</sub>	-9.2	0.8

Table 11: Influence of varying reversal cycle configuration on *K2* ratio measurement. Measurements are carried out with CMI measuring system. Notations E<sub>5</sub>, C<sub>10</sub>, A<sub>24</sub> and B<sub>340</sub> refer to Table 4.

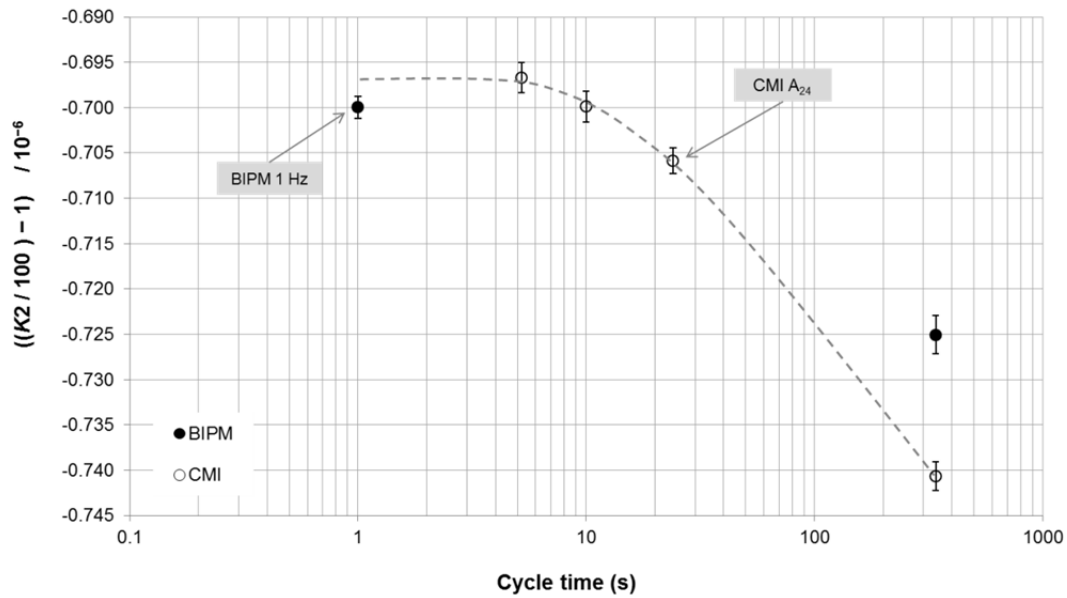


Figure 6: Differences from nominal value of  $K2$  ratio measured by BIPM and CMI for different polarity reversal frequencies. The  $K2$  value of BIPM for the cycle time of 340 s corresponds to the 1 Hz measurement corrected from the 1 Hz - dc error determined at the BIPM prior to the comparison (see section 3.2). Error bars correspond to standard combined uncertainties. Interpolated dotted line is just guide for the eyes.

The results of figure 6 are very similar to those obtained during the comparison with the PTB [2, 3]. No 'dc value' of ratio  $K2$  can be extrapolated for long cycle time measurements as no convergence toward a stable ratio value is observed, and a significant difference between BIPM and CMI is measured for the reference cycle time of 340 s. This difference has a relative value of about  $1.5 \times 10^{-8}$ . It is clearly higher than the resolution of both measuring systems and is of the same order of magnitude as that reported in [2]. It has been attributed to the quite different reversal timing schemes of the CCC current signals used by the two institutes in comparison (at 340 s), which would produce quite different thermal behaviors in the 1  $\Omega$  resistor. Further investigations on the effect of the reversal timing scheme would be necessary to confirm this hypothesis.

Another point that has been evidenced in [2, 3] is the existence of a plateau for short cycle times. It may be explained by the fact that below a given value, the cycle time becomes smaller than the time constant of the thermal emf induced by the Peltier effect [15]. In this previous comparison the plateau was found to begin for a cycle time of about 10 s, which is very similar to what we measured in the present case, figure 6.

## 7.2. Comparability of BIPM and CMI measurements of $K2$

From the above observations it is clear that it wouldn't be satisfactory to perform a comparison of the supposed 'dc' values of ratio  $K2$  determined by CMI and BIPM for a 340 s cycle time as the 'true dc value' cannot be estimated with a reasonable uncertainty (in regard to the resolution of the measuring systems). Nevertheless, the equivalence of the measuring systems of CMI and BIPM can still be demonstrated if we consider measurements of  $K2$  obtained for operating conditions being equivalent for both systems.

We suggest that the best equivalence of operating conditions for this comparison of  $K2$  was for cycle times corresponding to measured ratio values belonging to the above mentioned 'plateau', outside of which the

Peltier error starts to induce a significant error on  $K2$  measurements. Consequently, it was decided to compare  $K2$  ratio measurement of the BIPM at 1 Hz to that of the CMI at 5.2 s cycle time, those two measurements being on the plateau.

### 7.3. Influence on the $K2$ comparison uncertainty budget

When the 1 Hz-bridge of the BIPM is no longer used as a transfer instrument referenced to its CCC bridge, one has to take into account the uncertainty associated with the accuracy of its room temperature current comparator and resistive divider [8]. The uncertainty budget for the use of the BIPM 1 Hz-bridge for the measurement of the ratio  $K2$  is reported table 12.

Furthermore, in order to cover the assumptions that the plateau corresponding to a negligible Peltier effect is reached at a cycle time of 5.2 s and that the plateau begins for the same cycle time when using square or sinusoidal cycle shapes, a conservative relative standard uncertainty component of  $u_{\text{Peltier}} = 2.5 \times 10^{-9}$  was estimated.

Resistance ratio $K2$ ( $100 \Omega / 1 \Omega$ )	
Relative standard uncertainties	/ $10^{-9}$
<i>Ratio error of the room temperature current comparator</i>	1.0
<i>Resistive divider calibration of the secondary current source</i>	0.5
<i>Finite gain of servo of the bridge balance</i>	0.5
<b>Combined uncertainty, <math>u_B</math>=</b>	<b>1.2</b>

Table 12: Uncertainty budget associated with the BIPM 1 Hz-bridge for the measurement of the  $K2$  ratio.

### 7.4. BIPM and CMI measurements of $K2$

BIPM and CMI measurement results of the  $K2$  ratio carried out on 28 April 2017 are reported in tables 13 and 14. The two standards were connected alternately to the BIPM and CMI bridges and five BIPM measurements were interleaved with four CMI measurements. As already mentioned, these measurements were carried out in the same operating conditions as for the measurement of  $K1$  but with a nominal rms current of 50 mA in the  $1 \Omega$  resistor.

Table 13 includes BIPM results for cycle times of 1 s (1 Hz sine wave) and 340 s (reversal cycle of figure 2). Each of the BIPM measurements at 1 Hz is the mean value of 8 individual measurements, corresponding to a total integration time of about 40 minutes. The associated dispersion corresponds to the standard deviation of the mean of the 8 measurements. The 'dc' corrected  $K2$  values correspond to the 1 Hz values on which 1 Hz-dc correction of table 1 has been applied.

Table 14 contains CMI results for the standard 24 s cycle time and for the two additional 5.2 s and 340 s cycle times. Each of the CMI measurements for standard cycle time consists in 80 measurement cycles corresponding to an overall integration time of 33 minutes. The results reported for the 5.2 s and 340 s cycle times correspond to the standard measurement on which the  $A_{24}/E_5$  and  $B_{340}/A_{24}$  corrections of table 11 have been applied, respectively.

Figure 7 presents the series of measurement of the BIPM at 1 Hz and of the CMI at 5.2 s cycle time (from data of tables 13 and 14).



Time	$(K2_{\text{BIPM}}/100)-1$ / $10^{-6}$		Dispersion / $10^{-9}$
	Standard cycle time (1 s)	'dc' corrected (340 s)	
8:24	-0.700 53	-0.725 03	0.70
10:18	-0.699 71	-0.724 21	0.58
12:37	-0.699 49	-0.723 99	0.77
14:08	-0.699 01	-0.723 51	0.65
15:50	-0.701 04	-0.725 54	0.47
<b>Mean value =</b>	<b>-0.699 96</b>	<b>-0.724 46</b>	
<b>Standard deviation, <math>u_A</math> =</b>	<b><math>0.37 \times 10^{-9}</math></b>		

Table 13: BIPM measurements of the  $(100 \Omega / 1 \Omega)$  ratio  $K2$  for a cycle time of 1 s (1 Hz) and corrected measurements for 'dc' equivalent (340 s cycle of figure 2). Measurements carried out on 28 April 2017. Results are expressed as the relative difference from the nominal ratio value 100.

BIPM results for 1 Hz:  $K2_{\text{BIPM}} = 100 \times (1 - 0.699\,96 \times 10^{-6}) \Omega$

Relative standard uncertainty:  $u_{\text{BIPM}} = 1.3 \times 10^{-9}$

where  $u_{\text{BIPM}}$  is calculated as the quadratic sum of  $u_A = 0.37 \times 10^{-9}$  and  $u_B = 1.2 \times 10^{-9}$  (from table 12).

Time	$(K2_{\text{CMI}}/100)-1$ / $10^{-6}$			Dispersion / $10^{-9}$
	Standard 24 s cycle time 'A <sub>24</sub> '	5.2 s cycle time 'E <sub>5</sub> '	'dc corrected B <sub>340</sub>	
9:08	-0.705 80	-0.696 60	-0.740 60	0.74
11:20	-0.706 69	-0.697 49	-0.741 49	0.72
13:08	-0.705 81	-0.696 61	-0.740 61	0.64
14:46	-0.705 20	-0.696 00	-0.740 00	1.00
<b>Mean value =</b>	<b>-0.705 88</b>	<b>-0.696 68</b>	<b>-0.740 67</b>	
<b>Standard deviation, <math>u_A</math> =</b>	<b><math>0.31 \times 10^{-9}</math></b>			

Table 14: CMI measurements of the  $(100 \Omega / 1 \Omega)$  ratio  $K2$  for the standard cycle time ( $A_{24}$ ) and corrected measurements for 5.2 s and 340 s cycle times ( $E_5$  and  $B_{340}$  respectively). Measurements carried out on 28 April 2017. Results are expressed as the relative difference from the nominal ratio 100.

CMI results for 5.2 s cycle time:  $K2_{\text{CMI}} = 100 \times (1 - 0.696\,68 \times 10^{-6}) \Omega$

Relative standard uncertainty:  $u_{\text{CMI}} = 1.5 \times 10^{-9}$

where  $u_{\text{CMI}}$  is calculated as the quadratic sum of  $u_A = 0.31 \times 10^{-9}$ ,  $u_B = 1.2 \times 10^{-9}$  (from Table 3), and  $u_{\text{CT}} = 0.80 \times 10^{-9}$  the standard uncertainty on cycle time correction.

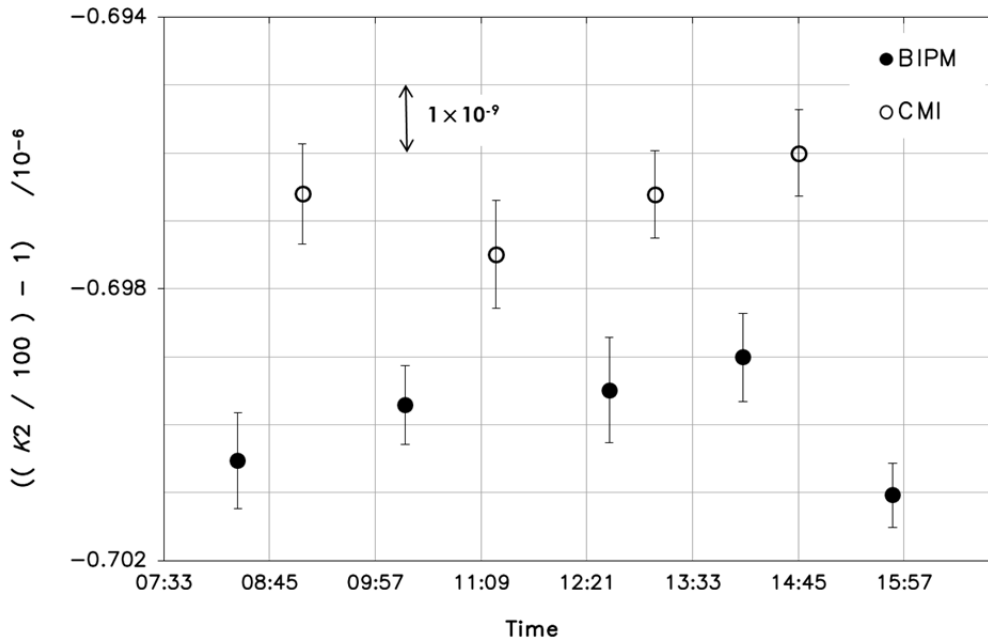


Figure 7: BIPM measurements at 1 Hz (black dots) and CMI corrected measurements at 5.2 s cycle time (white circles) of  $K2$  ratio. Measurements carried out on 28 April 2017 .

### 7.5. Comparison of $K2$ measurements

As stated in section 7.2, the best operating conditions of comparability of  $K2$  measurements consist in comparing the 1 Hz measurement of BIPM to the 5.2 s cycle time measurement of CMI. No significant instabilities of the standards were detected and therefore no additional uncertainty component was included in the final results.

The relative difference CMI - BIPM in the measurement of  $K2$  ratio was found to be:

$$(K2_{\text{CMI}} - K2_{\text{BIPM}}) / K2_{\text{BIPM}} = +3.3 \times 10^{-9}$$

Relative combined standard uncertainty:  $u_{\text{comp}} = 3.2 \times 10^{-9}$

where  $u_{\text{comp}}$  is calculated as the quadratic sum of  $u_{\text{BIPM}} = 1.3 \times 10^{-9}$ ,  $u_{\text{CMI}} = 1.5 \times 10^{-9}$  and  $u_{\text{Peltier}} = 2.5 \times 10^{-9}$  (see section 7.3).

## 8. Conclusion

The on-site key comparison BIPM.EM-K12 carried out in April 2017 between BIPM and CMI showed a very good agreement in the measurements of a conventional  $100 \Omega$  resistor in terms of the quantized Hall resistance ( $R_{\text{H}}(2)$ ), and in the determination of the resistance ratios  $K1$  and  $K2$  (ie.  $10\,000 \Omega/100 \Omega$  and  $100 \Omega/1 \Omega$ , respectively).

The results of the comparison are summarized in table 15. The relative difference between BIPM and CMI is of the order of 1 part in  $10^9$  or less for  $R_{100\Omega}$  and  $K1$ , and of about 3 parts in  $10^9$  for  $K2$ . Standard relative uncertainties are within 2.2 and 3.2 parts in  $10^9$ .

Additional measurements of the influence of cycle time (see sections 5.2.3, 6.2.2 and 7.1) have shown that careful attention must be paid to the current reversal period in order to obtain the best comparison results. Waiting time between reversals seems to be also an important parameter in the case of  $K2$  ratio measurements. It must be noticed that such observations were already made in previous works, the

references of which are mentioned in the above sections. In particular, the cycle time influence measured in the present comparison for ratio  $K2$  was very similar to that observed during a previous on-site comparison between BIPM and PTB [2].

Although it was always possible to find operating conditions in which the comparison of measuring systems from BIPM and CMI could be performed (i.e. for 'dc' equivalent reference 340 s cycle of the CCC-bridge of the BIPM for  $R_{100\Omega}$  and  $K1$ , and for 1 Hz and 5.2 s cycle times for  $K2$ ) a detailed explanation of the physical processes leading to the observed results would require a more thorough study of the influence of current reversals timing (including waiting times). This would help to have better criteria to define the so-called 'dc resistance' in future comparisons.

$R_{100\Omega}$ in terms of $R_H(2)$	$(R_{CMI} - R_{BIPM}) / R_{BIPM} = -0.6 \times 10^{-9}$	$u_{comp} = 2.5 \times 10^{-9}$
$K1 = R_{10k\Omega} / R_{100\Omega}$	$(K2_{CMI} - K2_{BIPM}) / K2_{BIPM} = +1.1 \times 10^{-9}$	$u_{comp} = 2.2 \times 10^{-9}$
$K2 = R_{100\Omega} / R_{1\Omega}$	$(K2_{CMI} - K2_{BIPM}) / K2_{BIPM} = +3.3 \times 10^{-9}$	$u_{comp} = 3.2 \times 10^{-9}$

Table 15: Summary of the results and associated relative standard uncertainties of the BIPM-CMI onsite comparison BIPM.EM-K12. The comparison measurements of  $K2$  were carried out at 1 Hz without 'dc' correction by the BIPM and with a cycle time of 5.2 s by the CMI.

The above results will also appear as Degree of Equivalence (DoE) in the BIPM Key Comparison Database (KCDB). The DoE of the participating institute with respect to the reference value is given by a pair of terms: the difference  $D$  from the reference value and its expanded uncertainty for  $k=2$ , i.e.  $U=2u$ . The reference value of the on-going comparison BIPM.EM-K12 was chosen to be the BIPM value.

The comparison results expressed as DoEs are summarized in table 16.

	Degree of equivalence $D / 10^{-9}$	Expanded uncertainty $U / 10^{-9}$
$R_{100\Omega}$ in terms of $R_H(2)$	<b>-0.6</b>	<b>5.0</b>
$K1 = R_{10k\Omega} / R_{100\Omega}$	<b>+1.1</b>	<b>4.4</b>
$K2 = R_{100\Omega} / R_{1\Omega}$	<b>+3.3</b>	<b>6.4</b>

Table 16: Summary of the comparison results expressed as degrees of equivalence (DoEs): difference from the BIPM reference value and expanded uncertainty  $U$  ( $k=2$ ).

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