BUREAU INTERNATIONAL DES POIDS ET MESURES

Bilateral comparison of 10 pF and 100 pF standards (ongoing BIPM key comparisons BIPM.EM-K14.a and 14.b) between the NMIM (Malaysia) and the BIPM

Final Report

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1 Introduction

This report presents the results of a bilateral comparison between the NMIM, Malaysia, and the BIPM, which was carried out using two 10 pF and two 100 pF travelling standards belonging to the BIPM. The comparison followed an *"A-B-A"* pattern of measurements during a total time of 19 weeks, from February 2022 to June 2022, and was piloted by the BIPM.

The measurand of this comparison was the two terminal-pair capacitance of the travelling capacitors at frequencies of 1000 Hz and 1592 Hz, and a measuring voltage of 15 V (rms) for the 10 pF standards and 10 V for the 100 pF standards.

This report covers the comparison of both 10 pF standards (BIPM.EM-K14.a) and 100 pF standards (BIPM.EM-K14.b) [1] as the two comparisons were carried out simultaneously, but they are analyzed separately.

2 Travelling standards

The four travelling standards used in this comparison are Andeen-Hagerling model AH11A capacitance modules having nominal values of 10 pF (SN 02213 and 02280), and 100 pF (SN 01574 and 02188), mounted together in a frame Andeen-Hagerling model AH1100 (SN 00200368).

The effect of ambient temperature on the standards mounted in the frame corresponds to their temperature coefficient, which is of the order of 1 part in 10^8/°C. According to the characterization of the travelling standards, performed previously at the BIPM, the effects of normal variations in humidity and atmospheric pressure were found to be negligible.

The voltage coefficient of capacitance and the relative change with frequency from 1000 Hz to 1592 Hz of the travelling standards were evaluated during the comparison at the BIPM, using as reference a 100 pF reference capacitor with known characteristics. Table 1 summarizes the results of these evaluations.

Table 1 Voltage coefficient of capacitance and relative change with frequency evaluation of the travelling standards of 10 pF (SN 02213 and 02280) and 100 pF (SN 01574 and 02188).

Capacitor	Voltage coefficient of capacitance ± standard uncertainty (pF / V)	Relative change with frequency ± standard uncertainty (nF/F)
10 pF SN 02213	$(0.0 \pm 2.0) \times 10^{-9}$	- 12 ± 35
10 pF SN 02280	$(+ 0.2 \pm 2.1) \times 10^{-9}$	- 15 ± 35
100 pF SN 01574	$(0.0 \pm 2.0) \times 10^{-7}$	+ 10 ± 35
100 pF SN 02188	$(+ 0.1 \pm 2.0) \times 10^{-7}$	+ 15 ± 35

Both laboratories measured the travelling standards within the environmental conditions summarized in Table 2.

Table 2 Environmental measurement conditions at BIPM and NMIM during the bilateral comparison.

Quantity	BIPM	NMIM
Temperature	(22.9 ± 0.3) °C	(23.4 ± 0.4) °C
Humidity	(50 ± 12) %	(47 ± 5) %
Atmospheric pressure	(1010 ± 16) hPa	(1005 ± 5) hPa

Due to the difference of ambient temperatures at BIPM and NMIM, an additional uncertainty contribution was included in the BIPM uncertainty budget.

The "DRIFT (PPM)" and "CHASSIS TEMP (°C)" indications of the AH1100 frame were recorded for completeness during all measurement periods, but they are not reported here, except for the relevant data for analysis of measurements for the 10 pF capacitor SN 02280.

3 Measurement principle

3.1 BIPM capacitance standard and measurement method.

The BIPM maintains its capacitance reference by measuring a group of fused silica capacitors twice a year, using a measurement chain linking the value of these capacitors to the value of the von Klitzing constant R_K, defined directly from the Planck constant h and the elementary charge e, according to:

$$R_K = h / e^2 = 25\ 812.807\ 46\ \Omega.$$

The mentioned measurement chain involves different measurement systems, such as a 10:1 ratio capacitance bridge, a multi-frequency quadrature bridge, a resistance bridge, an ac-dc coaxial resistor with calculable frequency dependence of resistance, and a quantum Hall device operated at 1 Hz [2, 3].

The repeated BIPM capacitance measurements were made at standard conditions of 1592 Hz and a voltage of 100 V (rms) for 10 pF and 10 V (rms) for 100 pF, using a 10:1 ratio capacitance bridge and 100 pF and 10 pF reference capacitors, respectively.

In order to match the frequency and voltage conditions of this comparison, previously fixed with the NMIM, the evaluated frequency coefficient was employed to correct the BIPM 10 pF and 100 pF measurements, while the evaluated voltage coefficient was used to correct exclusively the BIPM 10 pF measurements (see coefficients Table 1).

The BIPM measurements were performed during two periods of time, from 2 February 2022 to 14 March 2022 and from 6 May 2022 to 13 June 2022.

3.2 NMIM capacitance standard and measurement method.

Since 2003, the NMIM maintains the capacitance unit using a set of two 10 pF and one 100 pF Andeen-Hagerling AH11 type fused silica standard capacitors. These standards have been calibrated by the BIPM, with a two to three years periodicity. The most recent calibration of the standards was performed in September 2019, and intermediate checks were made regularly using a high-resolution digital capacitance bridge since 2008.

The NMIM reference values were determined by extrapolation using the results from all previous BIPM calibrations of the 10 pF capacitor SN 01112 and the 100 pF capacitor SN 01110, with support of the respective intermediate measurement results.

In order to match the voltage conditions of this comparison, the NMIM applied a voltage correction to the 10 pF capacitor SN 01112 value. This correction was made using a voltage coefficient of 0.9×10^{-9} pF/V, with a standard uncertainty of 2.0×10^{-9} pF/V, evaluated by the BIPM in September 2019.

The travelling standards were measured by a substitution method, using a digital capacitance bridge Andeen-Hagerling 2700 model option C as transfer standard.

The ambient temperature was kept at (23.4 ± 0.4) °C during the measurements and no temperature corrections were considered.

4 Measurement results

4.1 Comparison of 10 pF standards (BIPM.EM-K14.a).

The individual measurements performed at BIPM and NMIM for the 10 pF capacitors are shown on Figures 1 and 2 for the capacitor SN 02213, and Figures 3 and 4 for the capacitor SN 02280. On those Figures, it is reported the relative difference from nominal value (with C the measured capacitance value in pF) versus the measurement date for both frequencies 1000 Hz and 1592 Hz.

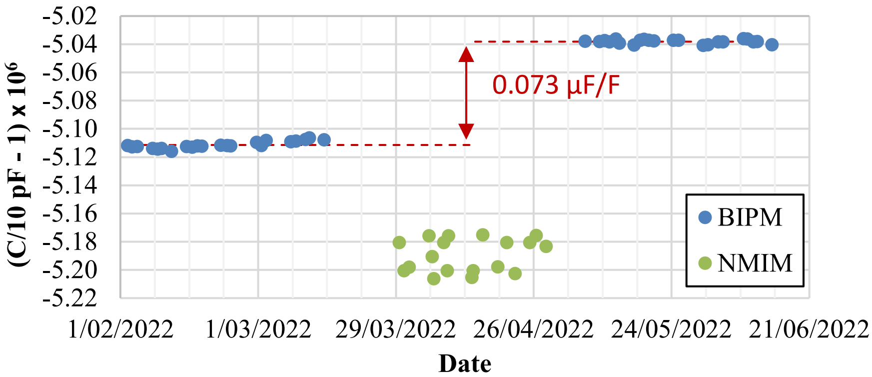

Figure 1 BIPM and NMIM measurements for the 10 pF capacitor SN 02213 at 15 V (rms) and 1000 Hz.

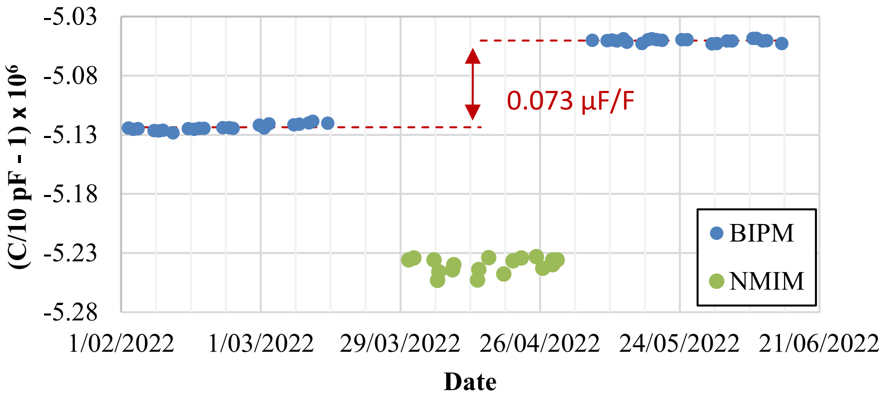

Figure 2 BIPM and NMIM measurements for the 10 pF capacitor SN 02213 at 15 V (rms) and 1592 Hz.

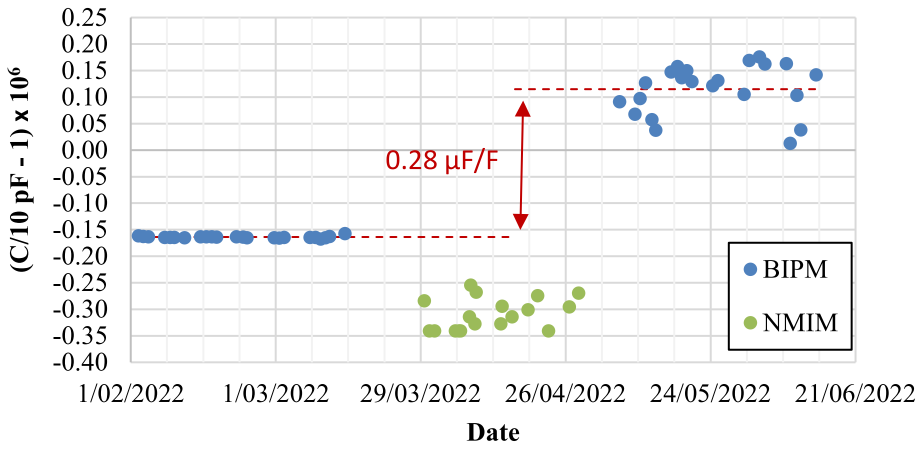

Figure 3 BIPM and NMIM measurements for the 10 pF capacitor SN 02280 at 15 V (rms) and 1000 Hz.

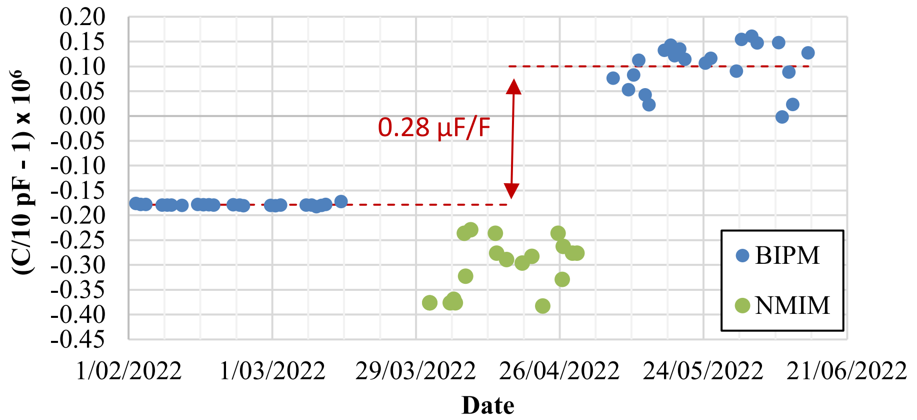

Figure 4 BIPM and NMIM measurements for the 10 pF capacitor SN 02280 at 15 V (rms) and 1592 Hz.

As can be seen, the capacitor SN 02213 behaved very well before and after the transport BIPM-NMIM-BIPM. The standard deviation for each of the two BIPM groups of measurements remained below 0.003 µF/F during at least 1 month. Additionally, it was identified that the value of this capacitor had a change of about 0.073 µF/F during the transport.

In the case of the capacitor SN 02280, the standard deviation of the first BIPM group of measurements remained below 0.003 µF/F, but after the transport BIPM-NMIM the capacitor became clearly unstable. After analyzing the corresponding “DRIFT (PPM)” front panel records, shown on Figure 5, and knowing that this indication is related with the temperature of the capacitive unit, according to the manufacturer, it was concluded that the temperature control of this capacitor was degraded or even damaged during the transport BIPM-NMIM. In addition, the value of the capacitor suffered an important change of about 0.28 µF/F during the transport. For these reasons, it was decided to exclude this capacitor from the comparison and to determine the reference value using only the results from the capacitor SN 02213.

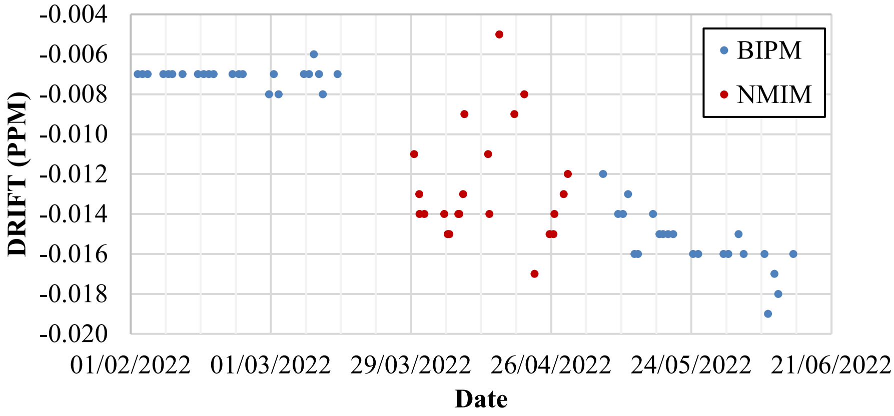

Figure 5 "DRIFT (PPM)" front panel readings of the 10 pF capacitor SN 02280.

After analyzing the BIPM and NMIM measurements for the capacitor SN 02213, and the computed ratios with the two 100 pF sets of measurements, it was not possible to determine if the change of the capacitor occurred during the transport BIPM-NMIM or NMIM-BIPM. For this reason, the reference value was estimated simply by using the average value of the BIPM measurements, and consequently, an additional uncertainty contribution was considered due to the transport, which was estimated considering that the reference value lies with equal probability within a uniform distribution having a width equal to 0.073 µF/F. Thus the estimated standard uncertainty associated with the transport was 0.021 µF/F, and it was included in the BIPM uncertainty budget.

The Table 3 shows the summary of the BIPM and the NMIM estimated values, and their corresponding 1σ standard uncertainties, for the 10 pF capacitor SN 02213. The detailed uncertainty budgets for BIPM and NMIM can be found in the annex of this report.

Table 3 Estimated value of capacitance for the 10 pF travelling standard SN 02213 at 15 V (rms) and frequencies of 1000 Hz and 1592 Hz.

	1000 Hz		1592 Hz	
Laboratory	**Capacitance**	**Standard uncertainty**	**Capacitance**	**Standard uncertainty**
BIPM	9.999 949 25 pF	0.061 µF/F	9.999 949 13 pF	0.050 µF/F
NMIM	9.999 948 1 pF	0.16 µF/F	9.999 947 6 pF	0.14 µF/F

The degree of equivalence D_{NMIM} between the BIPM and the NMIM, for the 10 pF standard at 15 V (rms) and frequencies of 1000 Hz and 1592 Hz, was computed using Equation 1, where C_{BIPM} is the capacitance value estimated by the BIPM, and C_{NMIM} is the capacitance mean value measured by the NMIM.

$$D_{\mathrm{NMIM}} = \frac{C_{\mathrm{NMIM}} - C_{\mathrm{BIPM}}}{C_{\mathrm{BIPM}}} \qquad (1)$$

The combined and expanded uncertainties associated with D_{NMIM}, u and U_{DNMIM} ($k = 2$, for a nominal confidence level of 95.45 %), respectively, were computed using the Equations 2 and 3.

$$U_{DNMIM} = 2\,u \tag{2}$$

$$u = \sqrt{u_{BIPM}^2 + u_{NMIM}^2} \tag{3}$$

For 10 pF at 15 V (rms) and 1000 Hz:

$\boldsymbol{D_{NMIM}}$ **= -0.12 μF/F**

$\boldsymbol{U_{DNMIM}}$ **= 0.34 μF/F**

For 10 pF at 15 V (rms) and 1592 Hz:

$\boldsymbol{D_{NMIM}}$ **= -0.15 μF/F**

$\boldsymbol{U_{DNMIM}}$ **= 0.30 μF/F**

4.2 Comparison of 100 pF standards (BIPM.EM-K14.b).

The individual measurements performed at BIPM and NMIM for the 100 pF capacitors at 1000 Hz and 1592 Hz are shown in Figures 6 and 7 for the capacitor SN 01574, and Figures 8 and 9 for the capacitor SN 02188. As for Figures 1 to 4, the relative difference from the nominal value (with C the measured capacitance value in pF) versus the measurement date is reported.

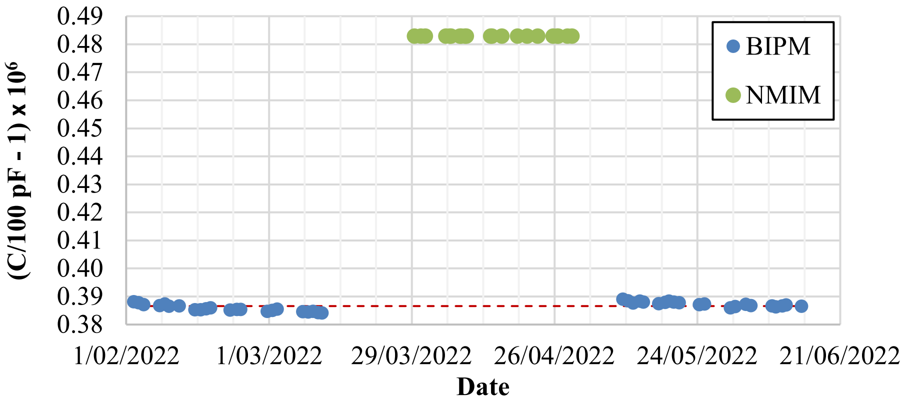

Figure 6 BIPM and NMIM measurements for the 100 pF capacitor SN 01574 at 10 V (rms) and 1000 Hz.

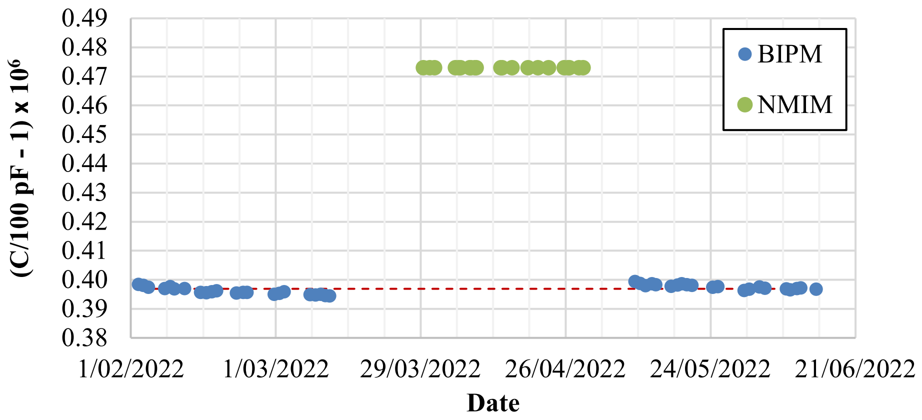

Figure 7 BIPM and NMIM measurements for the 100 pF capacitor SN 01574 at 10 V (rms) and 1592 Hz.

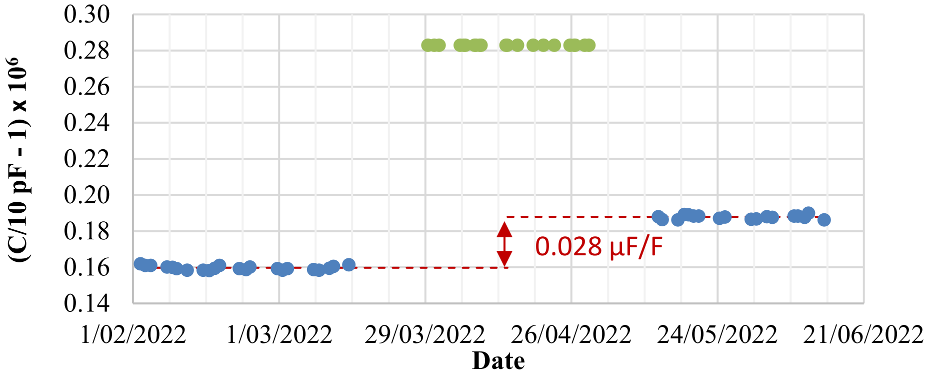

Figure 8 BIPM and NMIM measurements for the 100 pF capacitor SN 02188 at 10 V (rms) and 1000 Hz.

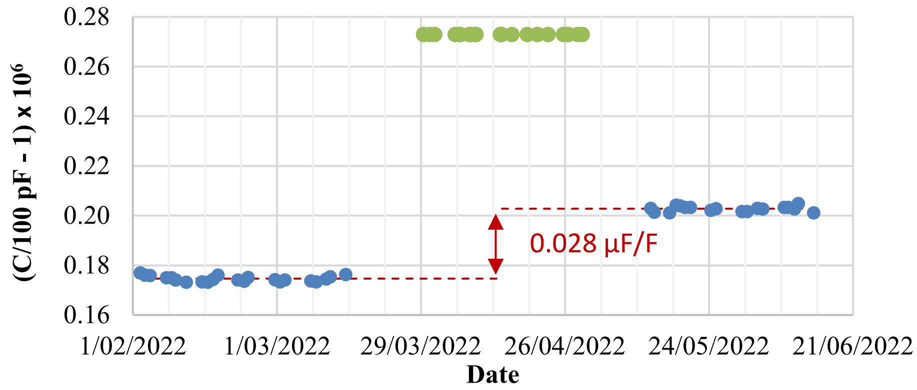

Figure 9 BIPM and NMIM measurements for the 100 pF capacitor SN 02188 at 10 V (rms) and 1592 Hz.

The capacitor SN 01574 behaved very well during the comparison, as can be seen in the corresponding Figures. The standard deviation for each BIPM groups of measurements is only 0.001 µF/F, and the difference between the average value of each group is only 0.002 µF/F. Taking into consideration these facts, the value for this capacitor was estimated simply by the average of all the measurements made at BIPM and the transport effect was considered negligible.

In the case of the capacitor SN 02188, the standard deviation for each BIPM groups of measurements is only 0.001 µF/F as well, but the value suffered a change of 0.028 µF/F during the transport BIPM-NMIM-BIPM. Taking advantage of the good stability of the 100 pF capacitor SN 01574, the ratio of measurements SN 02188 / SN 01574 was computed and analyzed to determine if the change of the capacitor occurred during the transport BIPM-NMIM or NMIM-BIPM. As can be seen in Figure 10, the NMIM computed ratio differs only 0.006 µF/F from the second group of BIPM measurements. Considering that the largest change of the capacitor occurred most likely during the transport BIPM-NMIM, it was decided to estimate the value of the capacitor SN 02188 using only the mean value from the BIPM measurements made after the transport NMIM-BIPM. As a consequence, it was included an additional uncertainty contribution to cover the transport effect. This uncertainty was estimated considering that the capacitor value lies with equal probability within a uniform distribution having a half width equal to 0.006 µF/F, thus the estimated standard uncertainty associated to the transport was 0.003 µF/F and it was included in the BIPM uncertainty budget.

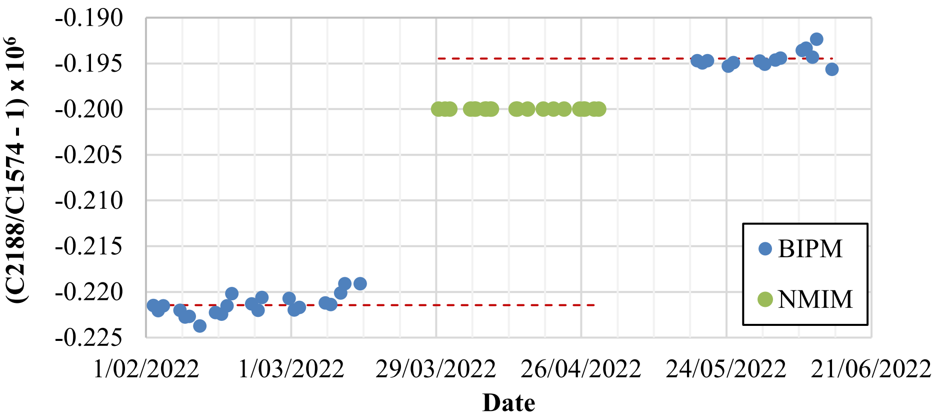

Figure 10 **Ratio between the measurements of the capacitors 100pF SN 02188 and 100 pF SN 01574.**

From the above, Tables 4 and 5 show the summary of the BIPM and the NMIM estimated values, and their corresponding 1σ standard uncertainties, for the 100 pF capacitors SN 01574 and SN 02213, respectively. The detailed uncertainty budgets for BIPM and NMIM can be found in the annex of this report.

Table 4 **Estimated value of capacitance for the 100 pF travelling standard SN 01574 at 10 V (rms) and frequencies of 1000 Hz and 1592 Hz.**

	1000 Hz		1592 Hz	
Laboratory	**Capacitance**	**Standard uncertainty**	**Capacitance**	**Standard uncertainty**
BIPM	100.000 038 7 pF	0.054 µF/F	100.000 039 7 pF	0.042 µF/F
NMIM	100.000 048 pF	0.21 µF/F	100.000 047 pF	0.20 µF/F

Table 5 Estimated value of capacitance for the 100 pF travelling standard SN 02188 at 10 V (rms) and frequencies of 1000 Hz and 1592 Hz.

	1000 Hz		1592 Hz	
Laboratory	**Capacitance**	**Standard uncertainty**	**Capacitance**	**Standard uncertainty**
BIPM	100.000 018 8 pF	0.054 µF/F	100.000 020 3 pF	0.042 µF/F
NMIM	100.000 028 pF	0.21 µF/F	100.000 027 pF	0.20 µF/F

The degree of equivalence D_{NMIM} between the BIPM and the NMIM, for the 100 pF standard at 10 V (rms) and frequencies of 1000 Hz and 1592 Hz, was computed using the Equation 4. $\overline{C_{\mathrm{BIPM}}}$ is the mean value of the BIPM estimations for the two traveling standards, and $\overline{C_{\mathrm{NMIM}}}$ is the mean value of the NMIM estimations for the two traveling standards.

$$D_{\mathrm{NMIM}} = \frac{\overline{C_{\mathrm{NMIM}}} - \overline{C_{\mathrm{BIPM}}}}{\overline{C_{\mathrm{BIPM}}}} \qquad (4)$$

The combined and expanded uncertainties associated with D_{NMIM}, u and U_{DNMIM} (k = 2, for a nominal confidence level of 95.45 %), respectively, were computed using the Equations 2 and 3.

For 100 pF at 10 V (rms) and 1000 Hz:

$$\boldsymbol{D_{\mathrm{NMIM}} = 0.09\ \mu\mathrm{F/F}}$$

$$\boldsymbol{U_{\mathrm{DNMIM}} = 0.43\ \mu\mathrm{F/F}}$$

For 100 pF at 10 V (rms) and 1592 Hz:

$$\boldsymbol{D_{NMIM} = 0.07\ \mu\mathrm{F/F}}$$

$$\boldsymbol{U_{DNMIM} = 0.41\ \mu\mathrm{F/F}}$$

4.3 Summary of results.

The degree of equivalence for the comparison of 10 pF and 100 pF standards (BIPM.EM-K14.a and BIPM.EM-K14.b respectively), are summarized in Table 6 and Figure 11.

Table 6 Degree of equivalence between the BIPM and the NMIM for 10 pF and 100 pF capacitance measurements and associated uncertainty (k = 2).

Nominal Value	1000 Hz		1592 Hz	
	D_{NMIM}	U_{DNMIM}	D_{NMIM}	U_{DNMIM}
10 pF	-0.12 µF/F	0.34 µF/F	-0.15 µF/F	0.30 µF/F
100 pF	0.09 µF/F	0.43 µF/F	0.07 µF/F	0.41 µF/F

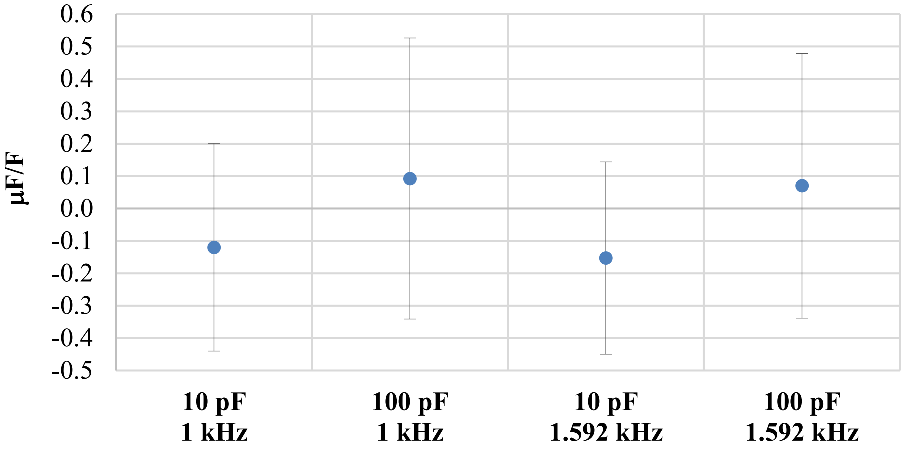

Figure 11 Degree of equivalence between the BIPM and the NMIM for 10 pF and 100 pF capacitance measurements and associated uncertainty (k = 2).

5 Comments and conclusion

The NMIM capacitance measurements are traceable to the quantized Hall resistance through periodic calibrations performed by the BIPM since 2003. Based on the calibration history and intermediate checks performed since 2008, the temporal drift effects of the reference standards have been well characterized, allowing consistent extrapolation values within the estimated uncertainty.

The BIPM.EM-K14.a and BIPM.EM-K14.b bilateral key comparisons between the NMIM and the BIPM were completed successfully, despite some transport effects and the misbehavior of one of the 10 pF traveling standards. The results obtained will be useful for the NMIM to support its current and future CMCs.

The most important identified uncertainty components in the comparisons are related to the NMIM extrapolation of the BIPM calibrations and to the use of the digital capacitance bridge, the latter supporting the surveillance of the standards through periodical intermediate checks, mainly for the 100 pF reference capacitor. These two uncertainty components contribute on average to about 80 % of the comparison’s uncertainty and 85 % of the overall NMIM measurement uncertainty.

Since there is no independent realization of the farad at the NMIM, the results of BIPM.EM-K14.a and BIPM.EM-K14.b comparisons only provide evidence to confirm that the drift of the NMIM capacitance references value is well considered, and the estimated uncertainty effectively reflects the actual state of the NMIM calibration system used to provide continuous calibration services to its clients.

References

[1] Protocol for BIPM on-going key comparisons of 10 pF and 100 pF capacitance standards (BIPM.EM-14a and BIPM.EM-K14b), BIPM publication.

[2] J. Angel Moreno and Pierre Gournay, 'Capacitance metrology at the BIPM to support National Metrology Institutes', 19th International Congress of Metrology, 2019, https://doi.org/10.1051/metrology/201914001

[3] F. Delahaye and R. Goebel, 'Evaluation of the frequency dependence of the resistance and capacitance standards in the BIPM quadrature bridge', IEEE. Trans. Instrum. Meas., 54, no 2, pp 533-537 (2005).

Annex - Uncertainty budgets

A.1 BIPM Uncertainty Budget for 10 pF measurements

The total uncertainty values reported in this Table correspond to the uncertainty on a single capacitance measurement at the BIPM. The uncertainties related to the stability of the capacitor during transport to NMIM and back, and to the effect of ambient temperature difference between BIPM and NMIM were also included.

Component	Relative uncertainty (10^{-9})	
	1000 Hz	**1592 Hz**
Values at 1 Hz of 51.6 kΩ resistors used in quadrature bridge, with respect to R_K.	14	14
1 Hz – 1541 Hz difference of 51.6 kΩ resistors.	22	22
Operation of quadrature bridge at 1541 Hz.	13	13
Scaling from 2000 pF capacitors of quadrature bridge to 10 pF or 100 references.	16	16
Time extrapolation of the value of the 10 pF or 100 pF references.	14	14
Link between traveling standards and 10 pF or 100 pF references.	20	20
Uncertainty on frequency correction (from 1592 Hz to 1000 Hz).	35	---
Uncertainty on voltage correction (change from 100 V to 15 V).	17	17
Repeatability of measurements.	3	3
Transport effect.	21	21
Difference of ambient temperatures at BIPM and NMIM.	5	5
Combined uncertainty at 15 V (rms)	**61**	**50**

All values are standard uncertainties (1σ estimates).

A.2 BIPM Uncertainty Budget for 100 pF measurements

The total uncertainty values reported in this Table correspond to the uncertainty on a single capacitance measurement at the BIPM. The uncertainties related to the stability of the capacitor during transport to NMIM and back, and to the effect of ambient temperature difference between BIPM and NMIM were also included.

Component	**Relative uncertainty (10^{-9})**	
	1000 Hz	**1592 Hz**
Values at 1 Hz of 51.6 kΩ resistors used in quadrature bridge, with respect to R_K.	14	14
1 Hz – 1541 Hz difference of 51.6 kΩ resistors.	22	22
Operation of quadrature bridge at 1541 Hz.	13	13
Scaling from 2000 pF capacitors of quadrature bridge to 10 pF or 100 references.	16	16
Time extrapolation of the value of the 10 pF or 100 pF references.	14	14
Link between traveling standards and 10 pF or 100 pF references.	20	20
Uncertainty on frequency correction (from 1592 Hz to 1000 Hz).	35	---
Repeatability of measurements	1	1
Transport effect.	3	3
Difference of ambient temperatures at BIPM and NMIM.	5	5
Combined uncertainty at 10 V (rms)	**54**	**42**

All values are standard uncertainties (1σ estimates).

A.3 NMIM Uncertainty Budget for 10 pF measurements

The uncertainty budget of the NMIM measurements is shown in the following Table.

Component	Relative uncertainty (10^{-6})	
	1000 Hz	**1592 Hz**
Reference Capacitor last BIPM calibration	0.040	0.040
Time extrapolation of BIPM calibrations	0.091	0.091
Transportation effects during last BIPM calibration	0.017	0.017
Frequency correction from 1592 Hz to 1000 Hz	0.060	---
Voltage correction from 100 V to 15 V	0.017	0.017
Reference Capacitor Temperature coefficient	0.002	0.002
Repeatability of measurements	0.001	0.001
Bridge resolution	0.003	0.003
Bridge uncertainty	0.100	0.100
Combined uncertainty at 15 V (rms)	**0.16**	**0.14**

All values are standard uncertainties (1σ estimates).

A.4 NMIM Uncertainty Budget for 100 pF measurements

The uncertainty budget of the NMIM measurements is shown in the following Table.

Component	**Relative uncertainty (10^{-6})**	
	1000 Hz	**1592 Hz**
Reference Capacitor last BIPM calibration	0.040	0.040
Time extrapolation of BIPM calibrations	0.165	0.165
Transportation effects during last BIPM calibration	0.046	0.023
Frequency correction from 1592 Hz to 1000 Hz	0.060	---
Reference Capacitor Temperature coefficient	0.002	0.002
Repeatability of measurements	0.000	0.000
Bridge resolution	0.029	0.029
Bridge uncertainty	0.100	0.100
Combined uncertainty at 10 V (rms)	**0.21**	**0.20**

All values are standard uncertainties (1σ estimates).