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 SASO (Saudi Arabia) and the BIPM

Final Report

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1 Introduction

This report presents the results of a bilateral comparison between the SASO, Saudi Arabia, 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 21 weeks, from October 2019 to February 2020, and was piloted by the BIPM.

The measurand of this comparison was the two terminal-pair capacitance of the travelling capacitors at a frequency of 1000 Hz, and a measuring rms voltage of 10 V.

This report covers the comparison of both the 10 pF standards (BIPM.EM-K14.a) and the 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 01227), 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 has been tested in the range 20 °C to 25 °C, within which no changes in the capacitance values greater than 1 part in 10^8 have been detected. Additionally, 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 01227) and 100 pF (SN 01574 and 02188).

Capacitor	Voltage coefficient of capacitance ± standard uncertainty (pF / V)	Relative change with frequency ± standard uncertainty (10^{-8})
10 pF SN 02213	(- 0.1 ± 2.0) x 10^{-9}	- 1.4 ± 6.1
10 pF SN 01227	(- 4.0 ± 2.0) x 10^{-9}	- 15.8 ± 6.1
100 pF SN 01574	(+ 0.1 ± 2.0) x 10^{-7}	+ 0.2 ± 6.2
100 pF SN 02188	(+ 0.1 ± 2.0) x 10^{-7}	+ 0.4 ± 6.2

Both laboratories measured the travelling standards within the following environmental conditions:

Temperature:	(23.3 ± 0.8) °C
Humidity:	(49 ± 15) %
Atmospheric pressure at BIPM:	(1005 ± 26) hPa
Atmospheric pressure at SASO:	(944 ± 3) hPa

The difference in atmospheric pressure is due to the fact that the laboratories are located at different altitudes, the BIPM at about 130 m above sea level and the SASO at about 610 m above sea level. Thanks to the fact that each individual travelling capacitor is hermetically sealed, any pressure correction to the capacitance values was not required.

The 'drift' and 'temperature' indications of the AH1100 frame were recorded for completeness during all measurement periods but are not reported here, apart from the relevant data regarding the analysis of measurements for the 10 pF capacitor SN 01227.

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:

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

This 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].

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

The evaluations of the voltage coefficient of capacitance and the relative change with frequency of the travelling standards, indicated in Table 1, were used to correct the BIPM measurements to match the conditions of 1000 Hz and 10 V of this comparison.

The BIPM measurements were performed during two periods of time, from 4 October 2019 to 4 November 2019 and from 21 January 2020 to 24 February 2020.

3.2 SASO capacitance standard and measurement method.

The SASO maintains the capacitance unit since 2015 by means of a set of four 10 pF and four 100 pF Andeen-Hagerling AH11A fused silica standard capacitors calibrated at UME-Turkey and NPL-UK. The most recent calibration of the 10 pF capacitors was provided by NPL-UK in December 2019, and for the 100 pF capacitors was provided by UME-Turkey in May 2017. Additionally, to maintain the value of the standards, intermediate checks are carried out periodically.

For this comparison, the value of the travelling standards was measured using a substitution method with a digital Capacitance Bridge AH 2700 as a transfer standard. The SASO reference value was defined using reference capacitors of the same nominal value, in particular two 10 pF capacitors with serial numbers 02080 and 02081, and two 100 pF capacitors with serial numbers 02084 and 02085, whose absolute values were computed from their calibration history. The measurements were carried out over a period of 1 month from 26 November 2019 to 25 December 2019, at an applied rms voltage of 10 V and a frequency of 1000 Hz.

4 Measurement results

To enable a better explanation of the measurement results obtained for the comparison of the 10 pF standards, the results for the comparison of the 100 pF standards will be explained first.

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

The individual measurements performed at BIPM and SASO for the 100 pF capacitors SN 01574 and 02188 are shown on Figures 1 and 2, respectively.

As can be seen, the capacitor 01574 behaved very well during all the comparison. The standard deviation of the two BIPM groups of measurements remained each below 0.002 µF/F during 1 month, and the difference between the average value of each group is only 0.003 µF/F. Taking these facts into consideration, the value for this capacitor was estimated by the average of all the measurements made at BIPM.

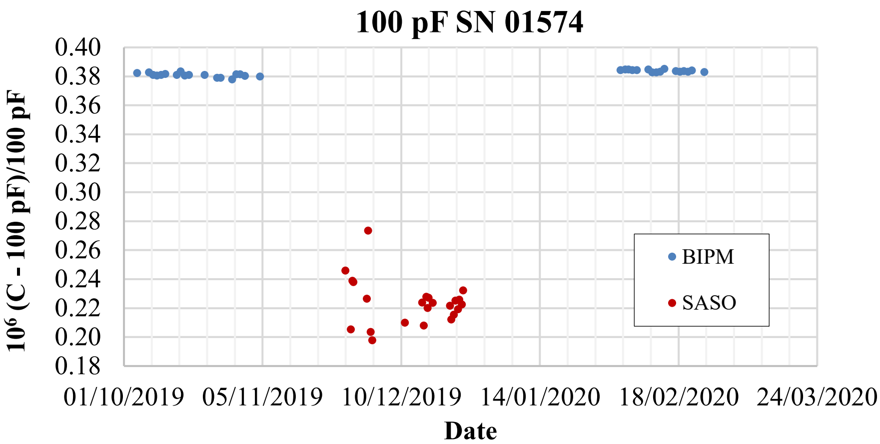

Figure 1 BIPM and SASO measurements for the 100 pF capacitor SN 01574.

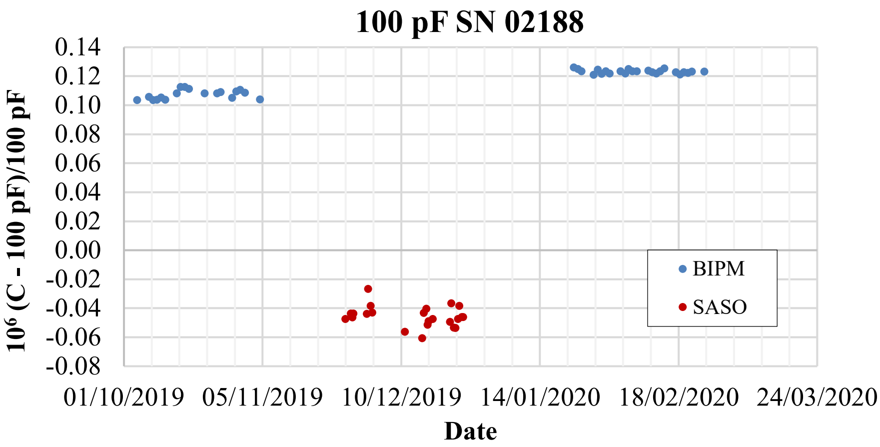

Figure 2 BIPM and SASO measurements for the 100 pF capacitor SN 02188.

In the case of the capacitor 02188, a small change of value was detected during the transport BIPM-SASO-BIPM. After inspection of the data, and thanks to the stability of the capacitor 01574, it was estimated that the change of the capacitor 02188 during the transport was +0.013 µF/F. Because the dispersion of the SASO measurements was higher than this change, it is not possible to know if the change occurred before or after the stay at the SASO. So, this change was considered as an additional uncertainty contribution due to transport, and the value of the capacitor was estimated using the average of the measurements made at BIPM.

Table 2 shows the summary of the BIPM and the SASO estimated values, as well as their corresponding standard uncertainties, for the 100 pF capacitors 01574 and 02188. The detailed uncertainty budgets for BIPM and SASO can be found in the annex of this report.

Table 2 Estimated value of capacitance for the 100 pF travelling standards SN 01574 and 02188 at 1000 Hz and 10 V (rms).

	Capacitor SN 01574		**Capacitor SN 02188**	
Laboratory	**Capacitance**	**Standard uncertainty**	**Capacitance**	**Standard uncertainty**
BIPM	100.000 038 2 pF	0.073 µF/F	100.000 011 6 pF	0.073 µF/F
SASO	100.000 022 pF	1.02 µF/F	99.999 995 pF	1.02 µF/F

After inspection of the values given in the Table 2, and the uncertainty budgets, it was concluded that the degree of equivalence D_{SASO} between the BIPM and the SASO for 100 pF standards at 1000 Hz and 10 V (rms) can be computed simply using equation 1, where $\overline{C_{\text{BIPM}}}$ is the mean capacitance value of the two capacitors measured by the BIPM, and $\overline{C_{\text{SASO}}}$ is the mean capacitance value of the two capacitors measured by the SASO.

$$D_{\text{SASO}} = \frac{\overline{C_{\text{SASO}}} - \overline{C_{\text{BIPM}}}}{\overline{C_{\text{BIPM}}}} \tag{1}$$

For both the BIPM and SASO, the uncertainty related to traceability is predominant and common for the measurement of the two capacitors. Its contribution to the uncertainty of the respective mean capacitance value are then expected to be fully correlated. Therefore the associated expanded uncertainty of D_{SASO}, U_{DSASO} (k = 2, for a nominal confidence level of 95.45 %), is computed with the equations 2 and 3, where u is the combined standard uncertainty computed by means of the estimated standard uncertainty of the BIPM, u_{BIPM}, and the standard uncertainty of the SASO, u_{SASO}.

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

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

D_{SASO} = -0.2 µF/F

$U_{D\mathrm{SASO}}$ = 2.1 µF/F

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

The individual measurements performed at BIPM and SASO for the 10 pF capacitors SN 02213 and 01228 are shown on Figures 3 and 4, respectively.

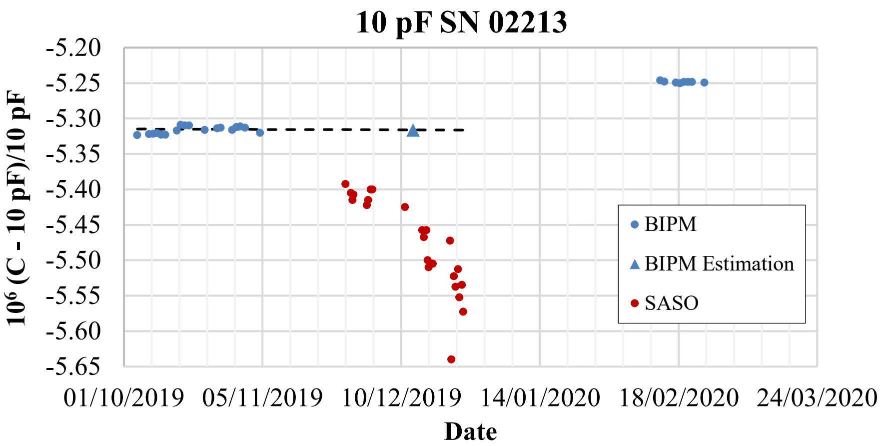

Figure 3 BIPM and SASO measurements for the 10 pF capacitor SN 02213.

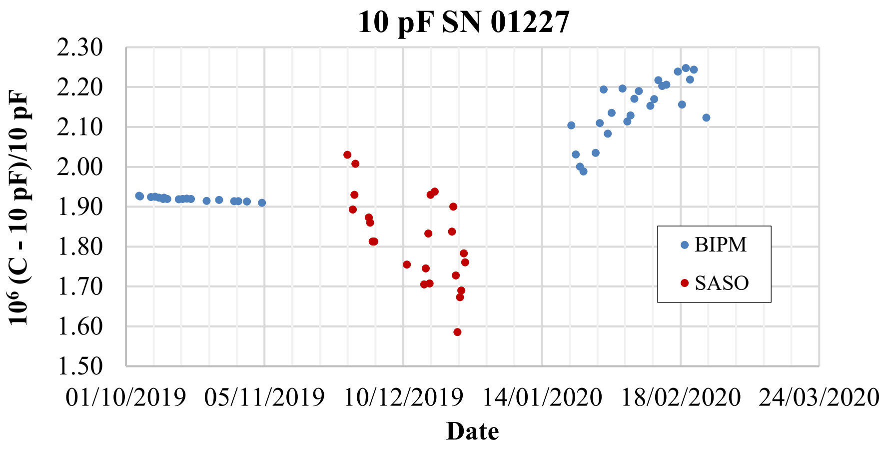

Figure 4 BIPM and SASO measurements for the 10 pF capacitor SN 01227.

In the Figure 3 it can be seen that the capacitor 02213 suffered a significant change of value during the transport BIPM-SASO-BIPM. From previous characterization, it is known that this capacitor had a very small drift of -0.002 µF/F during the four months previous to the transport BIPM-SASO. Using this information, it was estimated that the capacitor 02213 changed about +0.069 µF/F during the comparison. In order to find out if this change occurred before or after the stay at the SASO, the results of the comparison of 100 pF standards were used.

Taking advantage of the good stability of the 100 pF capacitor 01574, the ratio between the measurements of this capacitor and the measurements of the 10 pF capacitor 02213 was computed. As can be seen on Figure 5, the dispersion of the measurements made at the SASO is bigger than the change of the capacitor 02213, so it is not simple to find the moment at which the capacitor changed. However, analyzing the extrapolation trends from each group of measurements made at the BIPM, it can be seen that the trend of the first group of measurements, labeled as "Trend 1", lies closer to the average of the measurements made at the SASO, which means that the change of the capacitor 02213 could occur most likely during the transport SASO-BIPM.

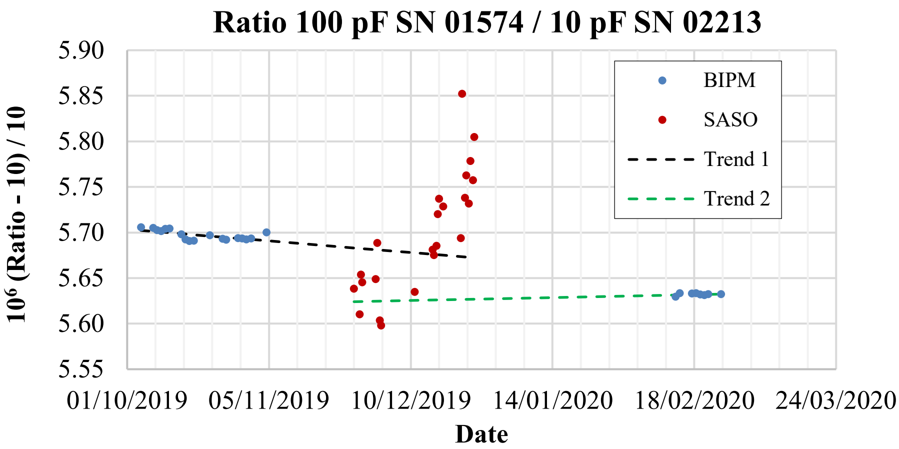

Figure 5 Ratio of capacitors 100pF SN 01574 / 10 pF capacitor SN 01227.

Even considering the above assumption, it is still possible that an additional jump occurred during the transport BIPM-SASO, which arises from the fact that the "Trend 1" still differs 0.051 µF/F from the mean value of the SASO measurements. It was concluded that the best estimation of the value of the capacitor 02213 can be obtained by means of the extrapolated value using the measurements made at the BIPM before the transport BIPM-SASO, as it is depicted in the Figure 3, with an additional uncertainty contribution due to transport effects coming from the possibility of the described additional jump.

For the capacitor 01227, different aspects of the measurements were analyzed.

a) As can be seen in the Figure 4, before the transport to the SASO, the BIPM measurements followed a linear trend with a predictable drift of about -0.015 µF/F per month. Under these conditions, the use of the capacitor is perfectly reasonable for the comparison.

b) After arriving to the SASO, the capacitor presented a completely different behavior, which seems to be induced by an external factor, as for instance:

   i. ambient conditions,
   ii. the SASO measurement system, or
   iii. a malfunction in the capacitive unit.

   Using the reported data, different possible correlations were investigated. The ambient quantities as temperature, humidity and pressure were not found to be correlated with the measured capacitance value. Furthermore, the standard deviation of the measurements of the capacitor 01227 was almost twice as big as that of capacitor 02213, so it was discarded that the origin of this behavior were located in the SASO measurement system. Finally, neither the chassis 'temperature' nor the 'drift' value of the capacitive unit revealed a definitive influence in the measurements.

c) After return at the BIPM, the capacitor presented again a different behavior, in this case with a poorly defined trend with erratic changes. Exactly as for the SASO measurements, different possible correlations were investigated. No external or measurement system effect was found to have an influence over the capacitance value, excepted for the 'drift' value. As can be seen in the Figure 6, the capacitance and the 'drift' value maintained a very strong correlation, with a coefficient of $r = -0.91$.

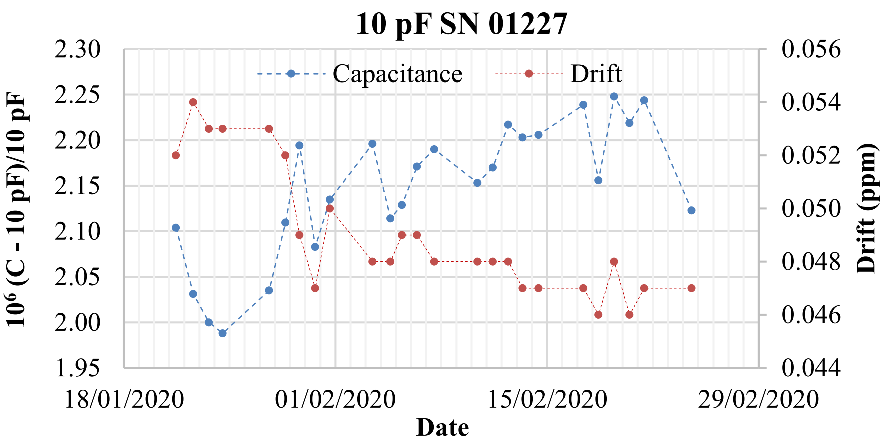

Figure 6 Correlation of the capacitance value and 'drift' of the 10 pF capacitor SN 01227.

According to the manufacturer, the 'drift' indication is related to the temperature of the capacitive unit, so it was assumed that the behavior of the capacitor most likely originated in a damage or failure of some electronic component or subsystem of the temperature control.

Taking into consideration the different elements of the above analysis, it is not possible to exclude that the capacitor 01227 suffered some damage since the transport BIPM-SASO. Thus, it was decided to exclude it from this comparison and to determine the BIPM-SASO difference using only the results obtained with the capacitor 02213.

The BIPM and the SASO estimated values and their corresponding 1σ standard uncertainties, for the capacitor 02213 exclusively, are summarized in Table 3. The detailed uncertainty budgets for BIPM and SASO can be found in the annex of this report.

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

Laboratory	Capacitance	Standard uncertainty
BIPM	9.999 946 84 pF	0.076 μF/F
SASO	9.999 945 3 pF	0.40 μF/F

The degree of equivalence D_{SASO} between the BIPM and the SASO for 10 pF standards at 1000 Hz and 10 V (rms) is computed as shows the equation 4, where C_{BIPM} is the capacitance value estimated by the BIPM, and C_{SASO} is the capacitance value measured by the SASO.

$$D_{SASO} = \frac{C_{SASO} - C_{BIPM}}{C_{BIPM}} \qquad (4)$$

The associated expanded uncertainty of D_{SASO}, U_{DSASO} (k = 2, for a nominal confidence level of 95.45 %), is computed with the equations 2 and 3.

D_{SASO} = -0.15 μF/F

U_{DSASO} = 0.81 μF/F

4.3 Summary of results.

The degree of equivalence of each of the comparison BIPM.EM-K14.a and BIPM.EM-K14.b of 10 pF and 100 pF standards respectively, are summarized in Table 4 and Figure 7.

Table 4 Degree of equivalence between the BIPM and the SASO for 10 pF and 100 pF capacitance measurements and associated uncertainty (*k* = 2).

Nominal Value	D_{SASO}	U_{DSASO}
10 pF	-0.15 μF/F	0.81 μF/F
100 pF	-0.2 μF/F	2.1 μF/F

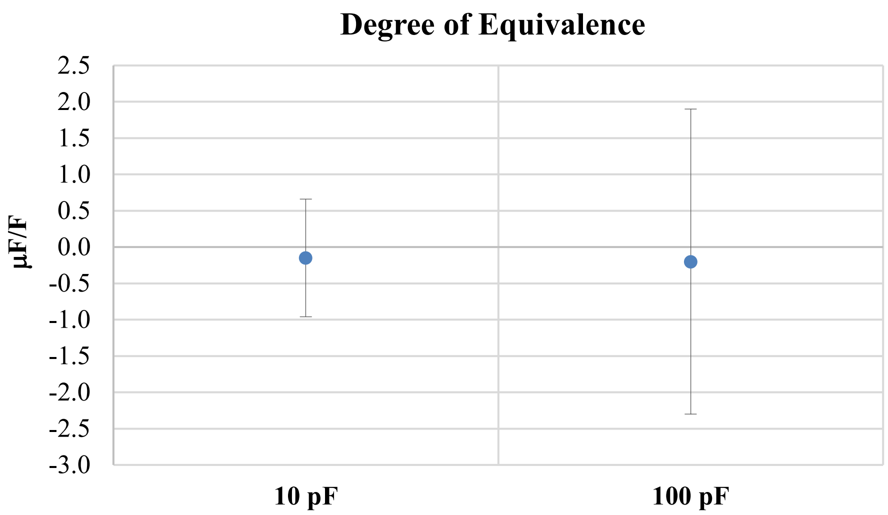

Figure 7 Degree of equivalence between the BIPM and the SASO for 10 pF and 100 pF capacitance measurements and associated uncertainty (*k* = 2).

5 Comments and conclusion

As mentioned before, the SASO capacitance measurements are traceable to the UME-Turkey through periodic calibrations since 2015, and more recently to the NPL-UK for the 10 pF standards. Based on calibration history and intermediate checks, the temporal drift effects of these reference standards have been estimated and corrected. Using their routine operational measurement procedure, the SASO performed the calibration of the travelling standards of this comparison using a transfer system based on a commercial capacitance bridge and a substitution method.

The uncertainty of measurement is dominated by the contribution of the calibration of the standard capacitors, which is in the order of 73 % for the 10 pF standards and 97 % for the 100 pF standards. The uncertainty contribution from the drift of the reference capacitors and the dispersion of the measurements represents about 25 % of the overall uncertainty of the 10 pF results, and only 3 % of the 100 pF results.

Because there is no independent realization of the farad at the SASO, the results only provide evidence to confirm that the drift of the SASO capacitance reference values is well corrected with low enough uncertainty to provide continuous calibration services to the SASO clients within the claimed uncertainty. In addition, the comparison provides objective evidence that the measurement procedures developed and employed by the SASO staff are appropriate to support these calibration services.

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] 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

The total uncertainty values reported in this table correspond to the uncertainty on a single capacitance measurement at the BIPM.

Component	Relative uncertainty (10^{-9})	
	10 pF	**100 pF**
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.	15	15
Extrapolation of the value of the 10 pF or 100 pF references.	14	14
Link between unknown and 10 pF or 100 pF references.	15	15
Uncertainty on frequency correction (from 1592 Hz to 1000 Hz).	61	62
Uncertainty on voltage correction (change from 100 V to 10 V).	18	---
Transport effect.	15	7
Combined uncertainty at 1000 Hz and 10 V rms	**76**	**73**

All values are standard uncertainties (1σ estimates).

A.2 SASO Uncertainty Budget

The uncertainty budget of the SASO measurements is shown in the following table for each of the travelling standard, according the following mathematical model used in this comparison, where C_x is the capacitance value of the unknown capacitor.

$$C_{X\text{-}Cal} = [\, C_{x\text{-}Meas} + \delta C_{X\text{-}Res} + \delta C_{X\text{-}Temp} \,]$$

$$- \{ [\, C_{R\text{-}Meas} - \delta C_{R\text{-}Res} \,] - [\, C_{R\text{-}Cer} + \delta C_{R\text{-}Drf} + \delta C_{R\text{-}Voltage} + \delta C_{R\text{-}Temp} \,] \} + \delta_{Stb}$$

Component	**Relative uncertainty (10^{-6})**	
	10 pF	**100 pF**
Certificated value of the Reference Capacitor, $C_{R\text{-}Cer}$	0.346	1.000
Voltage coefficient of the Reference Capacitor, $\delta C_{R\text{-}Voltage}$	0.052	0.000
Drift of the Reference Capacitor from the last calibration, $\delta C_{R\text{-}Drf}$	0.104	0.165
Measurement results of the Reference Capacitor using the capacitance meter, $C_{R\text{-}Meas}$	0.120	0.050
Measurement results of the DUT using the capacitance meter, $C_{x\text{-}Meas}$	0.110	0.050
Finite resolution of the capacitance meter measuring the Reference Capacitor, $\delta C_{R\text{-}Res}$	0.003	0.003
Finite resolution of the capacitance meter measuring the DUT Capacitor, $\delta C_{X\text{-}Res}$	0.003	0.029
Temperature coefficient of the Reference Capacitor, $\delta C_{R\text{-}Temp}$	0.006	0.006
Temperature coefficient of the DUT Capacitor, $\delta C_{X\text{-}Temp}$	0.006	0.006
Stability of measuring device between two measurements, δ_{Stb}	0.058	0.029
Combined uncertainty at 1000 Hz and 10 V rms	**0.404**	**1.02**

All values are standard uncertainties (1σ estimates).