

# **CCEM-K4.2017 Comparison**

## **Technical Protocol**

**Comparison of 10 pF Capacitance Standards**

**Optional comparison of 100 pF Capacitance Standards**

## 1. Introduction

The Mutual Recognition Agreement (MRA) drawn up by the Comité International des Poids et Mesures (CIPM) provides the framework within which National Metrology Institutes (NMIs) demonstrate the equivalence of their measurement standards. The technical basis of the CIPM MRA consists of international comparisons of standards for several key quantities identified by the different Consultative Committees (CCs) of the CIPM. These key comparisons are carried out by the CCs, the Bureau International des Poids et Mesures (BIPM) and the Regional Metrology Organizations (RMOs), usually at the lowest possible level of uncertainty. They most often imply a limited number of NMIs from different RMOs and are complemented with regional key comparisons within the RMOs.

Among the electromagnetic quantities the CCEM (Consultative Committee for Electricity and Magnetism) has identified capacitance, at a value of 10 pF, as one of those key quantities. As such it is regularly compared within the framework of the CCEM-K4 key comparison.

The last CCEM-K4 comparison was carried out between 1996 and 1999 and has involved ten NMIs from four RMOs and the BIPM. The travelling standards were two 10 pF capacitors belonging to the NIST, which was also the pilot institute of the comparison. A set of regional key comparisons carried out within EURAMET (European Association of National Metrology Institutes), SIM (Inter-American Metrology System) and APMP (Asia Pacific Metrology Program) has subsequently complemented the comparison CCEM-K4. The results of all these comparisons are reported in references [1] to [4].

During the 12<sup>th</sup> meeting of the Working Group on Low Frequency Quantities (WGLF) of the CCEM in March 2013 it was decided to repeat this comparison. The general principles of the comparison were discussed during the 13<sup>th</sup> WGLF meeting in March 2015.

This protocol describes in detail the principles, the quantities to be measured and the overall organization of CCEM-K4.2017, which shall take place in 2017. General rules for “Measurement comparisons in the CIPM MRA” detailed in the document CIPM-MRA-D-05 [5] will apply throughout the present comparison as well as the complementary recommendations of the “CCEM Guidelines for Planning, Organizing, Conducting and Reporting Key, Supplementary and Pilot Comparisons” [6].

## 2. Comparison principle

The comparison scheme adopted by the CCEM is that of a large-scale star-comparison consisting in carrying out simultaneously a large number of bilateral comparisons piloted by the BIPM. In this scheme each participating institute is requested to send its own capacitance standards to BIPM which are then all compared during the same period to the BIPM reference capacitance standards. The participating laboratories are in charge of measuring their own standards before and after measurement at the BIPM (participant-BIPM-participant measuring scheme). Initial and return measurements are then reported to BIPM which is in charge, with the support group, of the analysis and computation of the comparison results.

The benefit of this practice is to shorten the comparison time and to limit the impact of a problematic capacitor to one participant. This type of comparison scheme has already been used for the key comparison CCEM-K1 of 1  $\Omega$  and 10 k $\Omega$  in 1990 [7].

### 3. Institutes eligible for participating to the comparison

In accordance with the above mentioned document "Measurement comparisons in the context of the CIPM-MRA" [5], the new key comparison CCEM-K4.2017 is opened to National Metrology Institutes and Designated Institutes of member states of the BIPM (hereinafter referred to as the Institute) able to realize and maintain a representation of the farad based on the link between capacitance standards and the quantized Hall resistance through a quadrature bridge, or on a calculable capacitor standard. It is also assumed that the participating institute has an adequate measurement chain allowing it to measure the absolute 10 pF key capacitance value with a relative standard uncertainty below or equal to 10 parts in  $10^8$ .

In the case where the number of potential participants for the comparison would be too large, a limited number of institutes belonging to the different RMOs will be selected in order to keep the comparison manageable in a reasonable amount of time. A main criterion to fix this number is tied to the number of standards that BIPM will have to measure on the same short time period. It will depend of the number of standards that each of the institute plan to send to BIPM (see paragraph 4.1).

## 4. Travelling standards

### 4.1 General requirements

In the comparison scheme adopted (§.2) the travelling standards are those of the participating institutes. Each institute shall send at least two, but not more than three, 10 pF capacitors for measurement to the BIPM. This should reduce the risk that the results for an institute are invalidated by an excessive drift of one of the capacitance standards related to accidental mechanical or thermal shocks during transportation (our experience at BIPM is that several standard capacitors mounted in the same thermo-regulating frame may behave differently although they have been transported under the same conditions). Optionally, two 100 pF capacitors can be sent as well (see 4.2).

The capacitance standards sent to BIPM for measurement should be suitable for measurement at an uncertainty level of a few parts in  $10^8$  or less. The capacitance values should be close to the nominal values within 1 part in  $10^4$ . Currently, the preferred commercial thermo-regulated capacitor model is the Andeen-Hagerling type AH11A but, while its technical specifications are less good, the air-bath version of the General Radio type GR 1408 is still acceptable. Note that the AH11A capacitors should be send to the BIPM in their thermo-regulating frame type AH1100 and that adaptors GR/BNC or GR/BPO should be provided with the GR 1408 model.

Other types of thermo-regulated capacitors (home-made for instance) of technical characteristics at least similar to those of the AH11A type (reported in table 1) are obviously acceptable.

Stability	Temperature coefficient (when thermo-regulated)	AC voltage coefficient @ 1 kHz	Dissipation factor ( $\tan \delta$ @ 1kHz)
0.4 ppm/year	0.01 ppm/°C	0.003 ppm/V	$3 \cdot 10^{-6}$

Table 1: Main commercial specifications of a 10 pF Andeen-Hagerling AH11A capacitor (see <http://www.andeen-hagerling.com/ah11a.htm> for a complete specification list).

## 4.2 Quantities to be measured

The mandatory quantity to be measured in this comparison is the capacitance of a standard capacitor of nominal value 10 pF within less than 1 part in  $10^4$ . Nevertheless, considering that the scaling from the 10 pF key value to other capacitance values is also an important point, an additional optional value of 100 pF is possible for institutes that would be interested. If this option is retained by the institute, it is expected that a total of no more than four standards is sent at BIPM. For institute which plan to send Andeen-Hagerling AH11A capacitors, this correspond to one full AH1100 frame equipped with either two 10 pF capacitors and two 100 pF capacitors, or three 10 pF capacitors and one 100 pF.

In the case where specific information would be necessary to operate correctly the standard capacitor(s) sent at BIPM, they should be notified in writing and sent with the capacitor(s). In particular, for Andeen-Hagerling standards, the required supply voltage (magnitude and frequency) should be explicitly mentioned.

### 4.2.1- Measurand

The measurand is the two-terminal pair capacitance value at the front panel input sockets of the measured standard capacitor. Each institute should use its normal measuring method taking care of the possible corrections for the effect of the connecting cables. The participating institute should apply these corrections to their measurements (initial and return measurement series) and provide the BIPM with the corrected capacitance values. Nevertheless, in the case where these corrections are small enough, they can be considered as an uncertainty component.

In order for BIPM to be able to apply the required cable corrections on the capacitance measurements performed at BIPM, each participating institute should provide, for each of its standard capacitors, the value of the parallel input capacitances at the “low” and “high” terminals (capacitance between the standard capacitive element and its shield). If required for the computation of corrections, the capacitance, inductance and resistance of the internal cables (from front panel – inner side - to the standard capacitive element) should be also provided.

With a view of making directly comparable the measurement results reported by each of the participating institutes, it is asked to provide the capacitance measurements in the SI farad unit. This means that, for those institutes whose traceability is based on a quantized Hall resistance, the value of the von Klitzing constant to be used should not be  $R_{K-90}$  but the value obtained by the last CODATA fundamental constants adjustment [8]. This value is  $R_K = 25\,812.807\,4555\ \Omega$  with a relative uncertainty of  $2.3 \times 10^{-10}$ .

#### 4.2.2- Measurement voltage and frequency

The rms voltage values to be applied on the 10 pF and the optional 100 pF standard capacitors are preferably 100 V and 10 V, respectively. If the rms voltages applied to the standard capacitors are not those recommended, the value of the voltage coefficient of the standards should be communicated to the BIPM.

The required measurement frequency is 1592 Hz. If the capacitance values of the capacitors sent for measurement to BIPM have not been measured at this frequency, then the capacitance values reported in the measurement report should be the corrected values at 1592 Hz. The frequency dependence of this capacitor will be notified to the BIPM.

Nevertheless, an optional frequency value of 1233 Hz is possible for NMIs running their quadrature transfer at this frequency. In the case of a NMI reporting its measurements to the pilot institute at 1233 Hz only, those results will be corrected for a frequency of 1592 Hz using the frequency coefficient of the NMI's standard capacitors. This coefficient will be determined by BIPM during the series of measurements carried out at the BIPM. Corrections will be determined and applied only on 10 pF capacitance standards which is the key comparison value. Also, an additional uncertainty corresponding to that of the frequency coefficient determination will be applied to the NMI's reported value.

Table 2 indicates the mandatory and optional measurements proposed in this comparison.

	Capacitance	Voltage	Frequency
Mandatory measurement	10 pF	100 V	1592 Hz
Optional measurements	10 pF	100 V	1233 Hz
	100 pF	10 V	1592 Hz

Table 2: Mandatory and optional measurements of comparison CCEM-K4.2017.

#### 4.2.3- Environmental conditions

The ambient temperature and relative humidity should be  $(23 \pm 1)^\circ\text{C}$  and  $(50 \pm 10)\%$  respectively.

The ambient temperature and the temperature of the standard capacitor must be recorded for each measurement (chassis temperature and drift for an AH11A capacitance standard). The relative humidity and the atmospheric pressure shall also be recorded.

If the measurement carried out by the institute is not performed within the specified ambient temperature range, the possible influence on the measured capacitance value must be known and, if necessary, a correction applied to report the value corresponding to an ambient temperature of  $23^\circ\text{C}$ . If applicable, the temperature coefficient of the (thermo-regulated) standards versus ambient temperature shall be notified to BIPM in order to apply the eventually required corrections on the measurements performed at BIPM.

It is not expected that corrections due to relative humidity or atmospheric pressure of the capacitance value will be applied, but if this is the case, they should be applied on the capacitance values reported in the measurement report and notified to BIPM.

## 5. Organization of the comparison

### 5.1 Coordination

As decided by the CCEM, the BIPM is the pilot institute for CCEM-K4.2017. The comparison will be organized by the task group composed of the BIPM, the LNE and the NIST. Contact persons for each of these institutes are:

Pierre Gournay, BIPM (pilot institute): [pierre.gournay@bipm.org](mailto:pierre.gournay@bipm.org)

Yicheng Wang, NIST: [yicheng.wang@nist.gov](mailto:yicheng.wang@nist.gov)

Olivier Thévenot, LNE: [olivier.thevenot@lnr.fr](mailto:olivier.thevenot@lnr.fr)

### 5.2 Participants

8 institutes have responded positively to the official invitation to participate in CCEM-K4.2017. The list of the institutes, their RMOs and the details of the contact persons are given in annex 1.

### 5.3 Time schedule

Participating laboratories should carry out two set of measurements: the initial and the return sets. In between the travelling standards will be compared at the BIPM. Each of the three sets of measurements is expected to take five weeks during which at least ten measurements will be performed (typically two per week separated by 3 or four days). The sets of measurements are separated in time only by the transportation from the participating institute to the BIPM (or back) plus the required stabilization time of the capacitance standards following transportation (ideally at least one week). To reduce the influence of the drift of the BIPM reference standards as much as possible, it is highly desirable that all travelling standards would be at the BIPM at the same time.

Unless any subsequent change, the comparison time schedule will be as reported in annex 2.

### 5.4 Transport of the standards

The participating institutes are advised that the packaging and transport of their own capacitance standards should be carefully prepared to facilitate custom clearances and to avoid the deterioration or the loss of the standards during transportation.

The participating institutes shall make their own "door to door" arrangements and shall cover the cost of transport of their own standards to or from the BIPM. In the case that the BIPM has to make some arrangements for the shipment and customs clearance operations, the corresponding costs will be charged to the Institute.

For transport from inside the European Union, no custom documents are required. However, for countries outside the European Union, an appropriate license for temporary exportation or an ATA carnet should be established and accompany the capacitance standards.

Detailed instructions for sending the capacitance standards to the BIPM are provided in annex 3 (procedure BIPM/ADM-DOU-T-02 issued from the BIPM Quality Management System). It is important

that each participating institute scrupulously complies with those instructions in order to limit the transit period of the standards at customs at arrival and/or departure from the BIPM. These instructions are the usual ones followed during all regular bilateral comparisons with BIPM.

As explained in annex 3, institutes are asked to fill in the form BIPM/ADM-DOU-F-12 (also issued from the BIPM Quality Management System) to provide the BIPM with the relevant information for a smooth receipt and return of the standard capacitors. This form is reported in annex 4. It is important that it is filled in and returned to the BIPM prior to the departure of the standards from the participating institute to the BIPM.

The BIPM will not provide insurance cover for the capacitance standards in transit that belong to the comparison participants although, if requested, this can be arranged and charged to the participant.

### **5.5 Packing and handling of the capacitance standards**

The capacitance standards are sensitive to mechanical shocks and to a lesser extent to thermal ones. They should be packed in a properly padded transport case. It should include a packing list indicating the items contained in the case but also all needed information about the use of the standards and possible frequency, voltage and temperature dependences.

Whenever possible, the standard capacitors should be kept thermalized at the temperature of measurement during transportation. This requires that they are equipped with a battery having a sufficient capacity and a dc-ac converter to keep them powered during their travel. For those institutes having the possibility to organize the transportation by car, the dc-ac converter could be connected directly to the car battery through the 12 V socket. However, as such transportation may be difficult or impossible to organize for certain institutes, the choice to keep the standards thermalized during transportation is let to their responsibility.

Each participating institute should inform the BIPM of the departure of its standards using the dispatching note reported in annex 5 (after arranging the customs procedures, see 5.4). The BIPM will inform the institute of the arrival of its standards using the receipt note reported in annex 6.

After the completion of the measurements at BIPM all institutes will be informed of the departure of their standards from BIPM using the form of annex 5.

### **5.6 Failure of the travelling standards**

In the case where the travelling standards of an institute would arrive damaged at BIPM, the institute would be informed as fast as possible so that it may arrange the sending of another possibly available capacitance standard. Also, for the same reasons, if an abnormal drift or instability is observed on one or several standards after the first measurements, the institute will be quickly informed.

## 6 Measurement recordings

Each participating institute shall perform the capacitance measurements using their own usual measuring method traceable to either  $R_K$  through a Quantum Hall Resistance or to a Calculable Capacitor. A priori, this method should be the same as that declared in the best CMCs (Calibration and Measurement Capabilities); if not, it should be notified in the measurement report to be submitted by the institute at the end of the comparison.

For each capacitance measurement carried out on a given standard capacitor, the quantities reported below should be recorded using the spreadsheet template reported in annex 7.

Quantities to be recorded:

- the date of measurement
- the measurement frequency
- the applied voltage
- the ambient conditions in the vicinity of the standard capacitor: temperature, relative humidity and atmospheric pressure
- the temperature of the standard capacitor (temperature of the chassis and drift (ppm) for AH11A capacitors)
- the measurement result for the capacitance as defined in paragraph 4.2.1

In complement to the above quantities, it is requested to indicate in the spreadsheet the type A and B uncertainty values for the actual capacitance measurement as well as the combined uncertainty value. In addition, overall uncertainty of the measurement of the ambient conditions and standard capacitor temperature should be indicated. All the measurement spreadsheets (one per capacitance value and per frequency) should be appended to the measurement report of the institute.

## 7 Reporting uncertainty budget

As mentioned above, the measurement results should be reported together with their combined uncertainty. The detailed uncertainty budget comprising the statistic (type A) and systematic (type B) uncertainty components shall be reported for each nominal capacitance value and, if necessary, each operating frequency. Those uncertainty budgets should be prepared following the recommendations from the Guide to the Uncertainty in Measurement [9].

The uncertainty budget should be reported using the table template reported in annex 8. The contributions of the main uncertainty components expected to appear in this table are:

- Experimental standard uncertainty of a single capacitance measurement (type A)
- Uncertainty components relative to the primary standard used as reference for the measurement of the travelling standard (type B)



- Uncertainty components relative to the measuring bridges: main and auxiliary balances, current equalizers, injection network, auxiliary balance, detector noise and offset, .... (type B)
- Uncertainty on lead corrections (type B)
- Uncertainty due to frequency changes (and voltage if needed) (type B)
- Uncertainty due to temperature - and other ambient parameters if needed (type B)
- All additional uncertainty specific to the measurement system used by the institute

## 8 Measurement report due by the participating Institute

Each participating institutes shall write a measurement report within six weeks after the date of completion of the last period of measurement (second measurement period at the participating institute). This report shall include at least the following information:

- the description of the traceability chain used to link the capacitance value to the primary standard (QHR or calculable capacitor)
- the description of the travelling capacitance standards
- the additional measurements carried out (optional measurements summarized in table 2)
- the description of the measuring bridges and transfer standards involved
- the description of the measurement procedure
- the measurement results as indicated in paragraph 6
- the complete uncertainty budget as indicated in paragraph 7

The report may be provided to BIPM either in the form of a signed paper version or in electronic form. In any case, the spreadsheet files containing the result of the measurements (template annex 7) should be provided with the report.

In order to allow a quick analysis of the results and the drafting of the Draft A report, it is important that each participating institute provides in its report at least all the information listed above, in a timely way. In the case where the report of an institute would not be received within a reasonable time its results will not appear in the Draft A of the comparison report and the institute will be de facto excluded from the comparison.

## 9 Comparison report

Drafting of the comparison reports (Drafts A and B) is the responsibility of the pilot laboratory with the support of the task group appointed by the CCEM. Draft A will include a summary of the information given by the participating institutes (see paragraph 8), differences between national standards, degrees of equivalence and all other useful information and analysis. Analysis of the data will rely on the usual methods used in key comparisons which are well documented in references [5,6,10 to 13].

Draft A of the comparison report will be submitted to the participating institutes within a few weeks following the reception of the last measurement report. This version of the report is strictly confidential. The participating institutes will be asked to return their comments on Draft A within four weeks after the date of its reception.

All comments except those concerning document formatting will be circulated to all participants and will be discussed until a consensus of views is reached. Draft B of the comparison report will be then prepared and submitted to WGLF and CCEM for approval. Draft B is not confidential and will become the final report once approved.

### 9.1 Method for comparing the results

The measurement results obtained during this comparison will be analyzed according to the basic principle that a set of  $N$  simultaneous bilateral comparisons have been carried out,  $N$  corresponding to the number of participating institutes including the BIPM. In effect, the BIPM will also measure one of its own sets of standards following the same procedure and time schedule as that established for the institutes. Since these comparisons will be performed using  $N$  different sets of transfer capacitance standards, the BIPM will serve as a common reference. The group of reference capacitors of the BIPM and its traceability are described in annex 9.

We present below a possible way to analyze the results of the comparison measurements. This will not necessarily be the final analysis scheme, but it should be applicable if the actual comparison measurements obtained do not stray too far from an ideal case.

As explained in paragraph 2, each of the participating institutes will perform the initial and return series of measurements on its own standards. The BIPM will carry out a series of measurements of these standards against its group of reference capacitors in between. For each institute, the reference capacitance value will be then defined as the value measured by the BIPM at the mean date of its measurement period.

The reference capacitance value will be compared to the value obtained by the institute, extrapolated from the initial and return series of measurements at the mean measurement date at BIPM. From our knowledge of the normal ageing of fused silica capacitors we should expect linear drifts (at least on the short duration of the comparison) allowing simple linear least square fittings of institutes' data sets. Nevertheless, bad transportation conditions (due to improper packing or handling during transportation) or instabilities after turning back the power on the standards after transportation (thermalization instabilities), may lead to step changes in the capacitance value of the standards and the linear fitting may become inappropriate and even impossible. In the event of this unexpected situation, it would be analyzed with the concerned institute.

Below are detailed the successive calculation steps followed to determine the difference between the reference value and the institute value. The index  $i$  is used to differentiate between the institutes and the index  $j$  is the number of the measurement in a series of  $n$  measurements carried out at the institute or at BIPM.

For the bilateral comparison of the institute  $i$  with BIPM we have at our disposal, for a single standard, three series of measurement results corresponding to the data sets,

- $(D_{i,j}^{Init}; C_{i,j}^{Init})$  with  $u_i^{Init}$  the standard uncertainty on  $C_{i,j}^{Init}$ , for the initial series at the institute,
- $(D_{i,j}^{BIPM}; C_{i,j}^{BIPM})$  with  $u_i^{BIPM}$  the standard uncertainty on  $C_{i,j}^{BIPM}$ , for the middle series at BIPM,
- $(D_{i,j}^{Return}; C_{i,j}^{Return})$  with  $u_i^{Return}$  the standard uncertainty on  $C_{i,j}^{Return}$ , for the return series at the institute,

where the notation  $D$  stands for the date of the measurement and  $C$  for the capacitance measurement (corrected for all necessary effects and in particular from cable influence). A possible example of graphical representation of these series is given on Figure 1.

The reference capacitance value  $C_i^{Ref}$  is calculated from the linear least squares interpolation (line  $\mathcal{L}_{BIPM}$  on figure 1) of the set of data points  $(D_{i,j}^{BIPM}; C_{i,j}^{BIPM})$  at the mean date  $\overline{D}_i^{BIPM} = \frac{1}{n} \sum_{j=1}^n D_{i,j}^{BIPM}$ .

The standard uncertainty associated with  $C_i^{Ref}$  is given by  $u(C_i^{ref}) = \sqrt{\frac{s_{BIPM}^2}{n} + (u_i^{BIPM})^2}$ , where  $s_{BIPM}^2$  is the estimator of the variance of the interpolated capacitance value  $C_i^{ref}$  at the date  $\overline{D}_i^{BIPM}$ , and  $u_i^{BIPM}$  is the standard uncertainty of a single capacitance measurement  $C_{i,j}^{BIPM}$ .

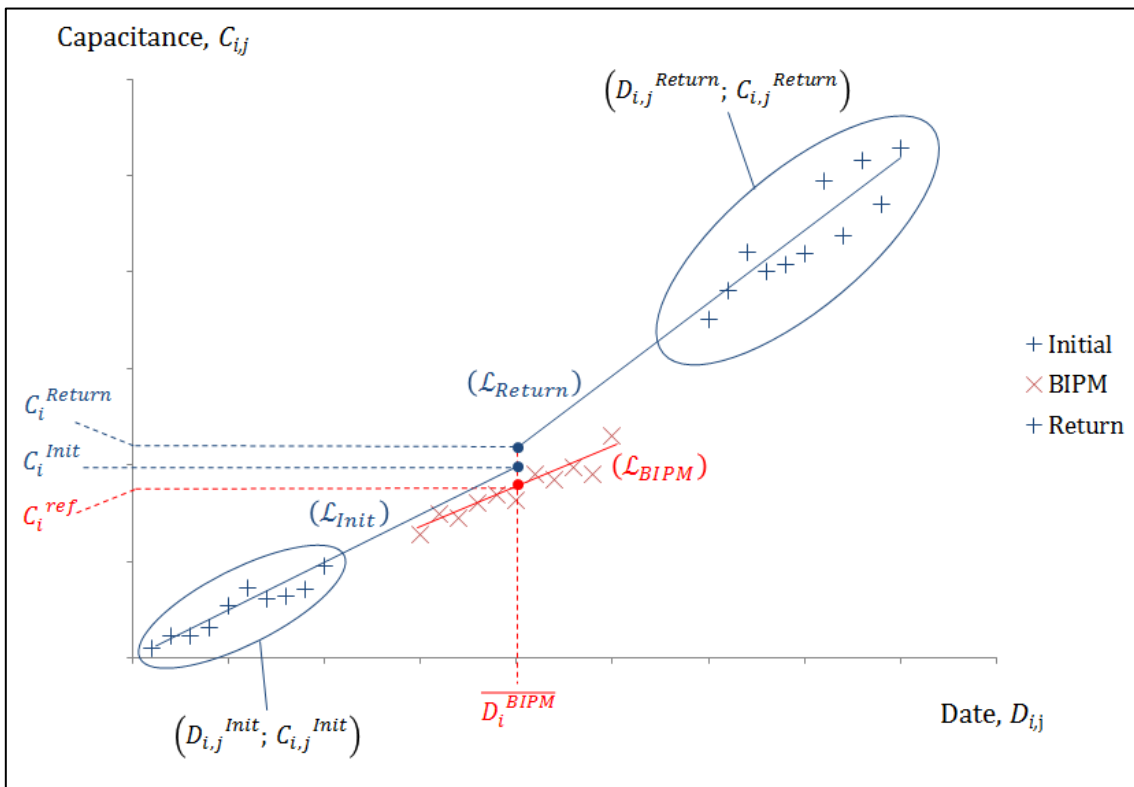


Figure 1: Example of graphical representation of the three series of measurements carried out by the institute  $i$  and by the BIPM (Initial series - BIPM series - Return series).

In a similar way, capacitance values  $C_i^{Init}$  and  $C_i^{Return}$  obtained by the institute  $i$  may be calculated from the linear least squares extrapolations ( $\mathcal{L}_{Init}$  and  $\mathcal{L}_{Return}$ ) of both the *Initial* and *Return* sets of data points ( $D_{i,j}^{Init}; C_{i,j}^{Init}$ ) and ( $D_{i,j}^{Return}; C_{i,j}^{Return}$ ) respectively, at the same mean date  $\overline{D_i^{BIPM}}$ .

Both the value of  $C_i^{Init}$  and  $C_i^{Return}$  correspond to predicted values extrapolated outside the range of dates in which the standard was measured. The combined uncertainties with which they are associated can be estimated from the quadratic sum of,

- the prediction uncertainty  $u_{pred}(C_i^{Init})$  or  $u_{pred}(C_i^{Return})$  obtained by applying the law of propagation of uncertainty on the equations of the fitting lines  $\mathcal{L}_{Init}$  or  $\mathcal{L}_{Return}$ ,
- and of the standard uncertainty of a single measurement  $u_i^{Init}$  or  $u_i^{Return}$  (for  $C_{i,j}^{Init}$  or  $C_{i,j}^{Return}$  respectively).

Thus, we will have,

$$u(C_i^{Init}) = \sqrt{u_{pred}^2(C_i^{Init}) + (u_i^{Init})^2} \quad \text{and} \quad u(C_i^{Return}) = \sqrt{u_{pred}^2(C_i^{Return}) + (u_i^{Return})^2}.$$

Notice that in an ideal case we should have identical linear interpolations for the initial and return measurement series at the institute and consequently  $C_i^{Init} = C_i^{Return}$ . Nevertheless, as suggested previously, such an ideal situation remains a priori hypothetical and possible drift slope changes or steps could be observed between initial and return series due to handling and transportation of the standards. This is why we propose to manage possible differences on  $C_i^{Init}$  and  $C_i^{Return}$  values as described above. For the same reason, the uncertainties  $u(C_i^{Init})$  and  $u(C_i^{Return})$  could be significantly different and the capacitance value  $C_i$  of the institute  $i$  to be compared to  $C_i^{Ref}$  is then considered as being the weighted mean of the above calculated values  $C_i^{Init}$  and  $C_i^{Return}$  using their own uncertainty as weight,

$$C_i = \frac{\frac{C_i^{Init}}{u^2(C_i^{Init})} + \frac{C_i^{Return}}{u^2(C_i^{Return})}}{\frac{1}{u^2(C_i^{Init})} + \frac{1}{u^2(C_i^{Return})}}$$

with the associated standard deviation,  $u_{wm}(C_i) = \left[ \frac{1}{u^2(C_i^{Init})} + \frac{1}{u^2(C_i^{Return})} \right]^{-\frac{1}{2}}$ .

This suggests also that we need to consider an uncertainty component  $u_{tr}(C_i)$  due to transportation of the standards. If the hypothesis is made that the value of  $C_i$  has a probability equal to 1 to be between  $C_i^{Init}$  and  $C_i^{Return}$  (rectangular distribution), an uncertainty component due to transportation may be defined as,

$$u_{tr}(C_i) = \frac{|C_i^{Init} - C_i^{Return}|}{2\sqrt{3}}.$$

Thus, the total uncertainty on  $C_i$  should then be,

$$u(C_i) = \sqrt{u_{wm}^2(C_i) + u_{tr}^2(C_i)} .$$

From the above determined values of  $C_i^{Ref}$  and  $C_i$  the difference between the institute  $i$  and the BIPM is simply given by,

$$\Delta_i = C_i - C_i^{ref} .$$

This difference is associated to the relative combined uncertainty,

$$u(\Delta_i) = \sqrt{u(C_i)^2 + u(C_i^{ref})^2}$$

with  $u(C_i^{ref})$  and  $u(C_i)$  defined as mentioned above.

In the case where the institute  $i$  would send a number  $p$  of standard capacitors of the same nominal value, the value of the difference  $\Delta_i$  for this institute will be calculated as the weighted mean of the  $p$  differences obtained for the  $p$  capacitors. The relative uncertainties  $u(\Delta_i)$  determined for each of the capacitors will be used in the calculation of the weights.

With  $q$  denoting the capacitance number and  $i$  the institute number, the weighted mean of the differences would be then,

$$\Delta_i = \frac{\sum_{q=1}^p w_q \Delta_{i,q}}{\sum_{q=1}^p w_q}$$

with the weights  $w_q$ ,

$$w_q = \frac{1}{u^2(\Delta_{i,q})} \quad \text{and} \quad u(\Delta_{i,q}) = \sqrt{u^2(C_{i,q}) + u^2(C_{i,q}^{ref})}$$

where  $\Delta_{i,q}$  is the difference between the measurements of the institute and the BIPM of  $C_{i,q}$ , the  $q^{\text{th}}$  capacitance standard of institute  $i$ .

The standard deviation associated to the weighted mean  $\Delta_i$  is then given by,

$$u(\Delta_i) = \left[ \sum_{q=1}^p w_q \right]^{-\frac{1}{2}} = \left[ \sum_{q=1}^p \frac{1}{u(\Delta_{i,q})^2} \right]^{-\frac{1}{2}} .$$

This calculation procedure is repeated for each of the  $N$  institutes involved in the comparison, that is to say for the  $N$  simultaneous bilateral comparisons carried out. We then obtain a set of  $N$  differences  $\Delta_i$  to the BIPM capacitance reference group of capacitors.

As mentioned at the beginning of this paragraph, BIPM will also, as all the other institutes, carry out the measurement of one of its own sets of standard capacitors following exactly the same measuring scheme. As a consequence, a difference between BIPM standards and BIPM reference group of

capacitors may similarly be calculated. This difference will correspond to  $\Delta_0$  and will have an associated uncertainty  $u(\Delta_0)$  whose value won't be impacted by the transportation uncertainty.

## 9.2 Key Comparison Reference Value (KCRV)

The BIPM is a participant in the comparison and the KCRV will not be defined to be equal to the BIPM reference value. If the comparison results are statistically well-behaved, which means that the dispersion of the results corresponds to the uncertainties, the KCRV will be calculated as the weighted mean value  $\bar{\Delta}$  of the differences  $\Delta_i$  between the institutes and the BIPM. The inverse of the squares of the relative uncertainties  $u(\Delta_i)$  associated with the  $\Delta_i$  will be used as the weights. Then,

$$\bar{\Delta} = \frac{\sum_{i=0}^{N-1} w_i \Delta_i}{\sum_{i=1}^N w_i}$$

with,

$$w_i = \frac{1}{u(\Delta_i)^2}$$

The combined relative uncertainty of the KCRV corresponds to the standard deviation associated with  $\bar{\Delta}$ , and is given by,

$$u(\bar{\Delta}) = \left[ \sum_{i=0}^{N-1} \frac{1}{u(\Delta_i)^2} \right]^{-1/2}$$

If the spread of the results would be significantly larger than the uncertainties, the weighted mean would not be the first choice, but the solution would then depend on the detailed situation.

## 9.3 Degrees of equivalence

The degree of equivalence (DoE) of the differences  $\Delta_i$  obtained by each of the participating institutes allows defining to what degree this difference is consistent with the KCRV ( $\bar{\Delta}$ ).

The DoE is expressed quantitatively by two terms: the deviation of  $\Delta_i$  from the KCRV and its uncertainty at a 95 % level of confidence (coverage factor  $k=2$ ). Then, the DoE of the institute number  $i$  is formed as the pair  $(d_i, U(d_i))$  with,

$$d_i = \Delta_i - \bar{\Delta} \quad \text{and} \quad U(d_i) = 2 \times u(d_i)$$

$$\text{where, } u(d_i) = [u^2(\Delta_i) - u^2(\bar{\Delta})]^{1/2}$$

If a consistent subset of  $\Delta_i$  would need to be determined due to significant discrepancies between institutes, then the uncertainty  $u(d_i)$  would be given by,

$$u(d_i) = [u^2(\Delta_i) - u^2(\bar{\Delta})]^{1/2} \quad \text{for institutes of the consistent subset}$$

$$\text{or, } u(d_i) = [u^2(\Delta_i) + u^2(\bar{\Delta})]^{1/2} \quad \text{for other institutes}$$

## References

- [1] Final report: CCEM Comparison of 10 pF Capacitance Standards, A.M. Jeffery, March 2002, [http://kcdb.bipm.org/appendixB/KCDB\\_ApB\\_info.asp?cmp\\_idy=42&cmp\\_cod=CCEM-K4&prov=exalead](http://kcdb.bipm.org/appendixB/KCDB_ApB_info.asp?cmp_idy=42&cmp_cod=CCEM-K4&prov=exalead)
- [2] Final Report SIM.EM-K4  
[http://kcdb.bipm.org/AppendixB/KCDB\\_ApB\\_info.asp?cmp\\_idy=620&cmp\\_cod=SIM.EM-K4&page=](http://kcdb.bipm.org/AppendixB/KCDB_ApB_info.asp?cmp_idy=620&cmp_cod=SIM.EM-K4&page=)
- [3] Final Report EUROMET EM-K4 (EUROMET Project n°345)  
[http://kcdb.bipm.org/AppendixB/KCDB\\_ApB\\_info.asp?cmp\\_idy=104&cmp\\_cod=EUROMET.EM-K4&page=](http://kcdb.bipm.org/AppendixB/KCDB_ApB_info.asp?cmp_idy=104&cmp_cod=EUROMET.EM-K4&page=)
- [4] Final Report APMP.EM-K4.1  
[http://kcdb.bipm.org/appendixB/KCDB\\_ApB\\_info.asp?cmp\\_idy=607&cmp\\_cod=APMP.EM-K4.1&prov=exalead](http://kcdb.bipm.org/appendixB/KCDB_ApB_info.asp?cmp_idy=607&cmp_cod=APMP.EM-K4.1&prov=exalead)
- [5] Measurement comparisons in the context of the CIPM MRA - CIPM-MRA-D-05  
<http://www.bipm.org/fr/cipm-mra/cipm-mra-documents/>
- [6] CCEM Guidelines for Planning, Organizing, Conducting and reporting Key, Supplementary and Pilot Comparisons, <http://www.bipm.org/en/committees/cc/ccem/publications-cc.html#bibliography>
- [7] Report on the 1990 International Comparison of 1  $\Omega$  and 10 k $\Omega$  Resistance Standards at the BIPM, F. Delahaye, D. Bournaud and T.J. Witt, Metrologia, **29**, pp. 273-283, 1992
- [8] <http://physics.nist.gov/cgi-bin/cuu/Value?rk>
- [9] Guide to the Expression of Uncertainty in Measurement, JCGM 100:2008, <http://www.bipm.org/fr/publications/guides/gum.html>
- [10] Evaluation of measurements by the method of least squares, L. Nielsen, <http://www.bipm.org>
- [11] Evaluation of key comparison data, M.G. Cox, Metrologia, 39, pp. 589-595, 2002
- [12] The evaluation of key comparison data; determining the largest consistent subset, M.G. Cox, Metrologia, 44, pp. 187-200, 2007
- [13] Modern regression methods, T.P. Ryan, Wiley, New York, ISBN 0-471-52912-5, 1997





# **ANNEX 1**

## **LIST OF PARTICIPATING INSTITUTES**

Institute	Country	RMO	Contact			
			Name	Telephone	Fax	email
BIPM Bureau International des Poids et Mesures	International	NA	Pierre Gournay	+33 1 45 07 70 07	+33 1 45 07 70 07	<a href="mailto:pierre.gournay@bipm.org">pierre.gournay@bipm.org</a>
LNE Laboratoire National de Métrologie et d'Essais	France	EURAMET	Olivier Thévenot	+33 1 30 69 21 76	+33 1 30 69 12 34	<a href="mailto:olivier.thevenot@lne.fr">olivier.thevenot@lne.fr</a>
METAS Federal Institute of Metrology	Switzerland	EURAMET	Frédéric Overney	+41 58 387 02 96		<a href="mailto:frederic.overney@metas.ch">frederic.overney@metas.ch</a>
NIM National Institute of Metrology	China	APMP	Yan Yang	+86 10 6452 4512	+86 10 6421 8629	<a href="mailto:yangyan@nim.ac.cn">yangyan@nim.ac.cn</a>
NIST National Institute of Standards and Technology	United State of America	SIM	Yicheng Wang	+1 301 975 4278	+1 301 926 3972	<a href="mailto:yicheng.wang@nist.gov">yicheng.wang@nist.gov</a>
NMIA National Metrology Institute of Australia	Australia	APMP	Leigh Johnson	+61 2 8467 3529	+61 2 8467 3752	<a href="mailto:Heather.Johnson@measurement.gov.au">Heather.Johnson@measurement.gov.au</a>
NPL National Physical Laboratory	United Kingdom	EURAMET	Janet Belliss	+44 (0)20 8943 6294		<a href="mailto:janet.belliss@npl.co.uk">janet.belliss@npl.co.uk</a>
PTB Physikalisch Technische Bundesanstalt	Germany	EURAMET	Jürgen Schurr	0049 - 531 - 592 2114	0049 - 531 - 592 2105	<a href="mailto:Juergen.Schurr@ptb.de">Juergen.Schurr@ptb.de</a>
VNIIM D.I. Mendeleyev Institute for Metrology	Russia	COOMET	Yuri Semenov	-	-	<a href="mailto:semyup@gmail.com">semyup@gmail.com</a>

## **ANNEX 2**

### **TIME SCHEDULE**


	Beginning date	End date	Duration
Measurement by Institutes	27 <sup>th</sup> February 2017	31 <sup>th</sup> March 2017	5 weeks
Transport	1 <sup>st</sup> April 2017	21 <sup>th</sup> April 2017	3 weeks
Standards stabilization	22 <sup>nd</sup> April 2017	30 <sup>th</sup> April 2017	1 week
Measurement by BIPM	1 <sup>st</sup> May 2017	23 <sup>th</sup> June 2017	8 weeks
Transport	24 <sup>th</sup> June 2017	14 <sup>th</sup> July 2017	3 weeks
Standards stabilization	15 <sup>th</sup> July 2017	23 <sup>th</sup> July 2017	1 week
Measurement by Institutes	24 <sup>th</sup> July 2017	1 <sup>st</sup> September 2017	6 weeks
Measurement report of Institutes	2 <sup>nd</sup> September 2017	13 <sup>th</sup> October 2017	6 weeks
Comparison report (draft A)	14 <sup>th</sup> October 2017	11 <sup>th</sup> December 2017	8 weeks

It is important to note that due to the scheme of the comparison it will be essential to have all transfer standards at the BIPM **simultaneously within a relatively short period.**

## **ANNEX 3**

### **INSTRUCTIONS FOR METROLOGY INSTITUTES SHIPPING EQUIPMENT TO THE BIPM FOR COMPARISONS**

**(procedure BIPM/ADM-DOU-T-02 issued from QMS of BIPM)**

Procédures Administration / Instructions for metrology institutes shipping equipment to the BIPM for comparisons			
Author : Isabelle Andernack Laïla Dell'Oro	Date : 2013/12/10 Version : 2.1	Authorized : Sigrid Arlen	

# ***INSTRUCTIONS FOR METROLOGY INSTITUTES SHIPPING EQUIPMENT TO THE BIPM FOR COMPARISONS***

## **1 General Information**

- Equipment shipped to the BIPM for comparisons is subject to Customs' formalities, which vary according to the country of origin.
- Before shipping any material to the BIPM, the metrology institute shall complete the relevant parts of the form **BIPM/ADM-DOU/F-12**, and return it duly signed to the BIPM (fax: +33 1 45 07 70 99 or e-mail at [ldelloro@bipm.org](mailto:ldelloro@bipm.org)). The form should be received by the BIPM at least 2 weeks before shipment is planned.
- Parcels from countries other than the E.U. must be labelled as follows:

**BIPM - REGLEMENTATION SPECIALE - NE PAS DEDOUANER D'OFFICE**

and the metrology institute from which the equipment originates should give specific instructions to their carrier to contact the BIPM

[Contact: Administration, tel.: +33 1 45 07 70 29 fax: +33 1 45 07 70 99]

prior to clearing the instrument through Customs. The BIPM will then take the appropriate action to clear the equipment through French Customs.

- No Customs' operations are carried out on Saturdays or Sundays. The metrology institute should ensure that if their equipment is subject to Customs' formalities, it should arrive in France on a working day of the week preceding that planned for the comparison.
- Customs' operations for hand carried equipment may require processing by the BIPM. In this case, relevant costs will be charged to the metrology institute.


## **2 Customs' formalities**

### **2.1 Equipment arriving from a country within the E.U.:**

- There are no Customs' formalities. The metrology institute does not need to take further action.

### **2.2 Equipment arriving from a country outside the E.U.:**

- There are Customs' formalities. In order for the equipment to pass through Customs, the metrology institute is required to undertake one of the following procedures:
  - i. ship the equipment with an ATA carnet. This carnet is available through the Chamber of Commerce and Industry (or equivalent within your country, provided your country

Procédures Administration / Instructions for metrology institutes shipping equipment to the BIPM for comparisons			
Author : Isabelle Andernack Laïla Dell'Oro	Date : 2013/12/10 Version : 2.1	Authorized : Sigrid Arlen	

recognises this system) and is issued with one year validity. It simplifies the Customs' operations and avoids duties and taxes;

- ii. ship the equipment by diplomatic bag to the relevant Embassy in Paris (although this has the advantage of by-passing all Customs' formalities, it is unlikely that this process is available to all metrology institutes);
- iii. if neither of these procedures can be adopted, a temporary importation will be arranged by the forwarding agent of the BIPM (all sections of the form **BIPM/ADM-DOU/F-12** must then be completed) and the relevant costs will be charged to the metrology institute. For hand carried equipment this will include an appointment on arrival at the airport with the forwarding agent of the BIPM, on a working day.

### 3 Transport of equipment between Paris Airports and the BIPM

#### 3.1 Equipment arriving from a country within the E.U.:


- For equipment originating from a metrology institute within the E.U., it is expected that the metrology institute will arrange a door-to-door delivery.
- In the case of air transport, it is expected that the metrology institute will arrange for their carrier to transport the equipment to and from Paris airports and the BIPM.

#### 3.2 Equipment arriving from a country outside the E.U.:

- For those countries employing the ATA carnet system, it is expected that the metrology institute will arrange a door-to-door delivery. In the case of air transport, it is expected that the metrology institute will arrange for their carrier to transport the equipment to and from Paris airports and the BIPM. The relevant costs will be charged to the metrology institute.
- For hand carried equipment, the metrology institute will arrange its transport between Paris airports and the BIPM.
- Where a temporary importation has to be arranged, the BIPM via its forwarding agent will arrange and meet the transport of the equipment to and from Paris airports and the BIPM.

### 4 Insurance of equipment

- In all cases, organisation and payment of insurance for a visiting metrology institute's instrument remain the responsibility of the visiting metrology institute.

Procédures Administration / Instructions for metrology institutes shipping equipment to the BIPM for comparisons				
Author : Isabelle Andernack Laïla Dell'Oro	Date : 2013/12/10 Version : 2.1	Authorized : Sigrid Arlen	BIPM/ADM-DOU-T-02	

## 5 Return of equipment

- It is the responsibility of the metrology institute to make prior arrangements for the return of their equipment after the comparison. The BIPM should be informed of these arrangements using form **BIPM/ADM-DOU/F-12**.
- No shipment back to the metrology institute will be arranged by the BIPM in the absence of this form duly completed and signed.
- Part “4. Instructions for return” of the form BIPM/ADM-DOU/F-12 is not applicable for BIPM equipment.




## **ANNEX 4**

### **SHIPPING INFORMATION AND INSTRUCTIONS FORM FOR CAPACITANCE STANDARDS RETURN**

**(procedure BIPM/ADM-DOU-F-12 issued from QMS of BIPM)**

<b>Procédures Administration / Shipping instructions for comparisons</b>			
Authors : I. Andernack B. Perent	Date : 2012/09/12 Version : 1.1	Authorized : Brigitte PERENT	BIPM/ADM-DOU-F-12



### **1. SHIPPING INSTRUCTIONS FOR COMPARISONS**

• Name of the metrology institute:		
• Person to be contacted:		
• Address:		
• Tel.:	• Fax:	• e-mail:

2. ATA carnet: <input type="checkbox"/>	Diplomatic bag: <input type="checkbox"/>	Other case: <input type="checkbox"/>
-----------------------------------------	------------------------------------------	--------------------------------------

### **3. SHIPPING INFORMATION**

• Description of the equipment (copy of proforma invoice required):		
• Value of the equipment:	• Number of packages:	
• Gross weight:	• Net weight:	
• N° AWB (when available):	• Date AWB:	
• Name of the carrier:		
• Hand carried by air (if necessary):	• flight number*:	• Date:
* A copy of the flight ticket and passport is required for travellers coming from non European		
• Hand carried by other means of transportation (to specify):	• Date:	

### **4. INSTRUCTIONS FOR RETURN**

• Insurance: <input type="checkbox"/> Yes <input type="checkbox"/> No		
• Name of the carrier:		
• Tel.:	• Fax:	• e-mail:
• Your client number with the carrier:		

### **5. I agree to pay for all the costs related to Customs' formalities and transport of equipment.**

Date	Name and title	Signature

## **ANNEX 5**

### **CONFIRMATION NOTE OF DISPATCH**

## Confirmation note of dispatch

### CCEM-K4 comparison of 10 pF capacitance

*(This form must be sent by email the day of departure of the standard)*

**FROM:**

Institute : \_\_\_\_\_

Contact person : \_\_\_\_\_

Email : \_\_\_\_\_

Tel. : \_\_\_\_\_

**TO:**

Institute : \_\_\_\_\_

Contact person : \_\_\_\_\_

Email : \_\_\_\_\_

Tel. : \_\_\_\_\_

We confirm having sent the standards to \_\_\_\_\_ on \_\_\_\_\_ (yyyyy/mm/dd).

Shipped equipment: \_\_\_\_\_

\_\_\_\_\_

\_\_\_\_\_

Additional information: \_\_\_\_\_

\_\_\_\_\_

Date: \_\_\_\_\_ (yyyyy/mm/dd)

Signature: \_\_\_\_\_

# **ANNEX 6**

## **CONFIRMATION NOTE OF RECEIPT**

## Confirmation note of receipt

### CCEM-K4 comparison of 10 pF capacitance

*(This form must be sent by email the day of receipt of the standard)*

**FROM:**

Institute : \_\_\_\_\_

Contact person : \_\_\_\_\_

Email : \_\_\_\_\_

Tel. : \_\_\_\_\_

**TO:**

Institute : \_\_\_\_\_

Contact person : \_\_\_\_\_

Email : \_\_\_\_\_

Tel. : \_\_\_\_\_

We confirm having received the standards from \_\_\_\_\_ on \_\_\_\_\_ (yyyyy/mm/dd).

Received equipment: \_\_\_\_\_

\_\_\_\_\_

\_\_\_\_\_

Additional information: \_\_\_\_\_

\_\_\_\_\_

Date: \_\_\_\_\_ (yyyyy/mm/dd)

Signature: \_\_\_\_\_

# **ANNEX 7**

## **TABLE TEMPLATE FOR MEASUREMENT RECORDING**

Serial number of the standard capacitor: \_\_\_\_\_

Nominal value: \_\_\_\_\_ Measurement frequency: \_\_\_\_\_ Applied voltage: \_\_\_\_\_

Date (yyyy/mm/dd) and Time (hh:mm)	Ambient conditions			Temperature of the standard		Measurement results			
	Temperature (°C)	Relative Humidity (%)	Atmospheric Pressure (Pa)	Chassis Temperature (°C)	Drift (ppm)	Deviation from nominal (μF/F)	Type A uncertainty (μF/F)	Type B uncertainty (μF/F)	Combined uncertainty (μF/F)
1									
2									
3									
4									
5									
6									
7									
8									
9									
10									

Remarks:

- Deviation from nominal should be corrected from cable effects and, if necessary, from temperature or ambient conditions influence.
- Overall uncertainty of ambient and capacitance temperatures, of relative humidity and atmospheric pressure should be indicated in the measurement report
- The part of the table relative to the temperature of the standard is given for a AH11A capacitance standard. It should be adapted to the actual standard used.



## **ANNEX 8**

### **TABLE TEMPLATE TO REPORT UNCERTAINTY BUDGET**

### Uncertainty statement

Nominal capacitance value :

Frequency:

Voltage:

Quantity / $X_i$	Estimate / $x_i$ (mention unit)	Standard uncertainty / $u(x_i)$ (mention unit)	Probability distribution	Sensitivity coefficient / $c_i$ (mention unit)	Contribution to relative standard uncertainty / $u_i(C_x)$ ( $\mu\text{F}/\text{F}$ )	Degrees of freedom / $\nu_i$

Measurand value /  $C_x$  :

Combined standard uncertainty /  $u_c(C_x)$  :

Effective degrees of freedom /  $\nu_{eff}$  :

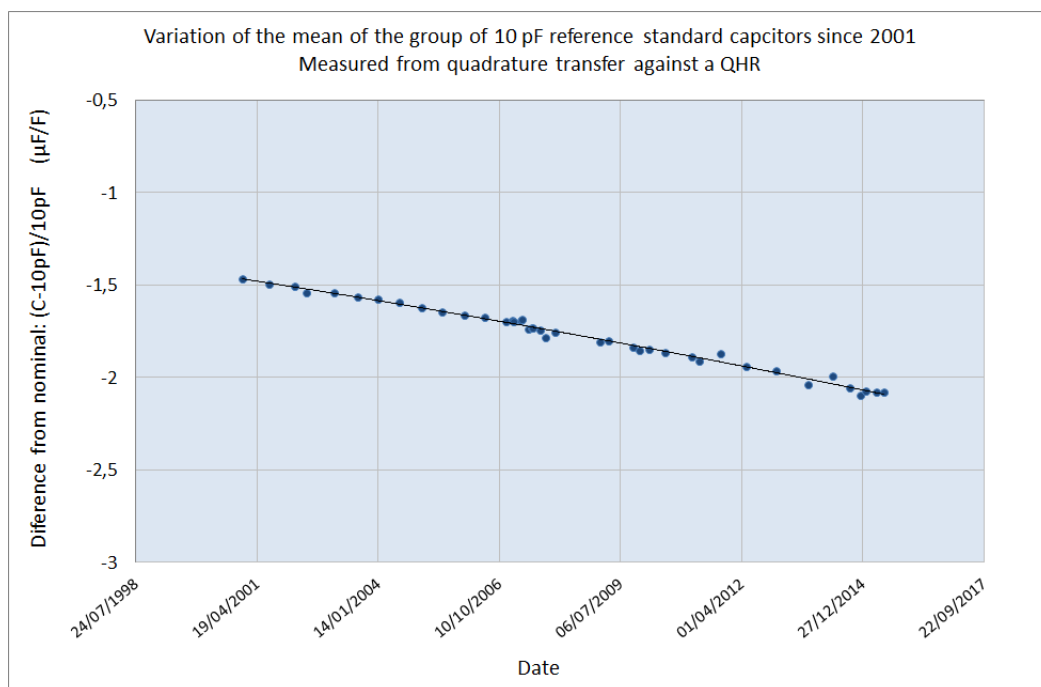
# **ANNEX 9**

## **10 pF CAPACITANCE REFERENCE GROUP OF BIPM**

The BIPM maintains a reference group of four fused silica 10 pF capacitors (one of the NBS type and three of the GR 1408-A type). The four capacitors are placed in a temperature-controlled oil bath at a nominal temperature of 25.00 °C. A platinum resistance thermometer of nominal value 25 Ω is permanently placed in the central well of each of the three GR 1408-A capacitors and the NBS one is equipped with a built-in platinum resistance thermometer, also of nominal value 25 Ω. The capacitance of each capacitor is by definition referred to a fixed conventional value of the corresponding thermometer resistance chosen to be close to the thermometer resistance at 25 °C. A correction is applied to the capacitance value at the time of measurement to take into account the difference between the measured thermometer resistance and the corresponding conventional value. This correction is calculated from the known temperature coefficients of each of the four capacitors.

Each capacitor of the group is equipped with two coaxial cables without current equalizer by which it is connected to a capacitance comparison bridge. Their capacitance is defined as the two terminal-pair capacitance at the end of the cables.

Since 2001, the mean value of the group has been measured very regularly (see figure below) using a measurement chain linking the 10 pF capacitances to the recommended value of the von Klitzing constant,  $R_{K-90} = 25\,812.807\ \Omega$ . The chain includes a two terminal-pair capacitance bridge with ratio 10/1, a multi-frequency quadrature bridge, an ac-dc coaxial resistor with calculable frequency dependence of resistance, and a quantum Hall device operated at 1 Hz. The relative drift rate of the mean value of the reference group is about 3.5 parts in  $10^8$  per year.



For the CCEM-K4 comparison, the mean reference group of capacitor will be calibrated against the QHR before and after the set of measurements performed at BIPM. The travelling standards of the participating Institute will be measured against the mean 10 pF capacitance of the group, directly on the 10:1 ratio bridge for the standards of optional 100 pF value, and via substitution (ie two 10:1 steps against a 100 pF buffer) in the case of the 10 pF standards.