Bilateral Comparison of 10 V Standards between the NSAI - NML (Ireland) and the BIPM,

February to March 2012

(part of the ongoing BIPM key comparison BIPM.EM-K11.b)

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Introduction

As part of the ongoing BIPM key comparison BIPM.EM-K11.b, a comparison of the 10 V voltage reference standards of the BIPM and the National Standards Authority of Ireland – National Metrology Laboratory (NSAI - NML)**,** Dublin, Ireland, was carried out from February to March 2012. Two BIPM Zener diode-based travelling standards (Fluke 732B), BIPM_C (ZC) and BIPM_D (ZD), were transported by freight to NSAI-NML. At NSAI-NML, the reference standard for DC voltage at the 10 V level consists of a group of characterized Zener diode-based electronic voltage standards. The output EMF (Electromotive Force) of each travelling standard was measured by direct comparison with the group standard.

At the BIPM the travelling standards were calibrated, before and after the measurements at NSAI-NML, with the Josephson Voltage Standard. Results of all measurements were corrected for the dependence of the output voltages on internal temperature and ambient atmospheric pressure.

Outline of the measuring method

NML-NSAI 10 V measurements

The EMF at the 10 V output terminals of the travelling standard is connected in series opposition to each individual member of the NSAI-NML group standard in turn, using a low thermal EMF scanner. The EMF differences are measured using a digital nanovoltmeter. The measured voltage differences, together with the predicted values of the NSAI-NML standards are subjected to a weighted least squares adjustment procedure in order to arrive at a best estimate of the unknown EMF.

The travelling standard is isolated from the mains supply during the measurements. The “GUARD” and “CHASSIS” terminals are jointly connected to a common ground point. The internal thermistor resistance is monitored during the measurements.

BIPM 10 V Measurements

The output voltage of the Zener standard to be measured is connected to the BIPM Josephson Voltage Standard (in series opposition with the BIPM array of Josephson junctions) through a low thermal EMF switch. The binding post terminals “GUARD” and “CHASSIS” of the Zener standard are connected together and connected to a single point which is the grounding reference point of the measurement setup.

The measurements start after at least two hours since the mains plug at the rear of the Zeners has been disconnected.

The BIPM detector consists of an EM model N1a analog nanovoltmeter whose output is connected, via an optically-coupled isolation amplifier, to a pen recorder and a digital voltmeter (DVM) which is connected to a computer.

This computer is used to monitor measurements, acquire data and calculate results. Low thermal electromotive force switches are used for critical switching, such as polarity reversal of the detector input.

After the BIPM array biasing frequency has been adjusted to a value where the voltage difference between the primary and the secondary voltage standards at nominally 10 V is below 0.5 µV, the nanovoltmeter is set to its 10 µV range to perform measurements. The measurement sequence can then be carried out. One individual measurement point is acquired according to the following procedure:

1- Positive array polarity and reverse position of the detector;

2- Data acquisition;
3- Positive array polarity and normal position of the detector;
4- Data acquisition;
5- Negative array polarity and reverse position of the detector;
6- Data acquisition;
7- Negative array polarity and normal position of the detector;
8- Data acquisition;
9- Negative array polarity and reverse position of the detector;
10- Data acquisition
11- Negative array polarity and normal position of the detector;
12- Data acquisition;
13- Positive array polarity and reverse position of the detector;
14- Data acquisition;
15- Positive array polarity and normal position of the detector;
16- Data acquisition;

The reversal of the array polarity (by inversing the bias current) is always accompanied by a reversal of the Zener voltage standard using a switch. The reversal of the detector polarity is done to cancel out any detector offset error and internal linear thermo-electromotive forces.

Each “Data Acquisition” step consists of 30 preliminary points followed by 500 measurement points. Each of these should not differ from the mean of the preliminary points by more than twice their standard deviation. The “Data Acquisition” sequence lasts 25 s. The total measurement time (including polarity reversals and data acquisition) is approximately 5 minutes.

This procedure is repeated three times and the mean value corresponds to one result on the graph (Cf. Fig. 1).

Results at 10 V

Figures 1a and 1b shows the measured values obtained for the two standards by the two laboratories at 10 V. Figure 2 presents the voltage evolution of the simple mean of the two standards.

A linear least squares fit is applied to the results of the BIPM to obtain the results for both standards and their uncertainties at the mean date of the NSAI-NML measurements (2012/02/23).

Note: The least square fit adjustment to the NSAI-NML data is only presented to illustrate the behavior of the standards but is not used for the calculation of the final result.

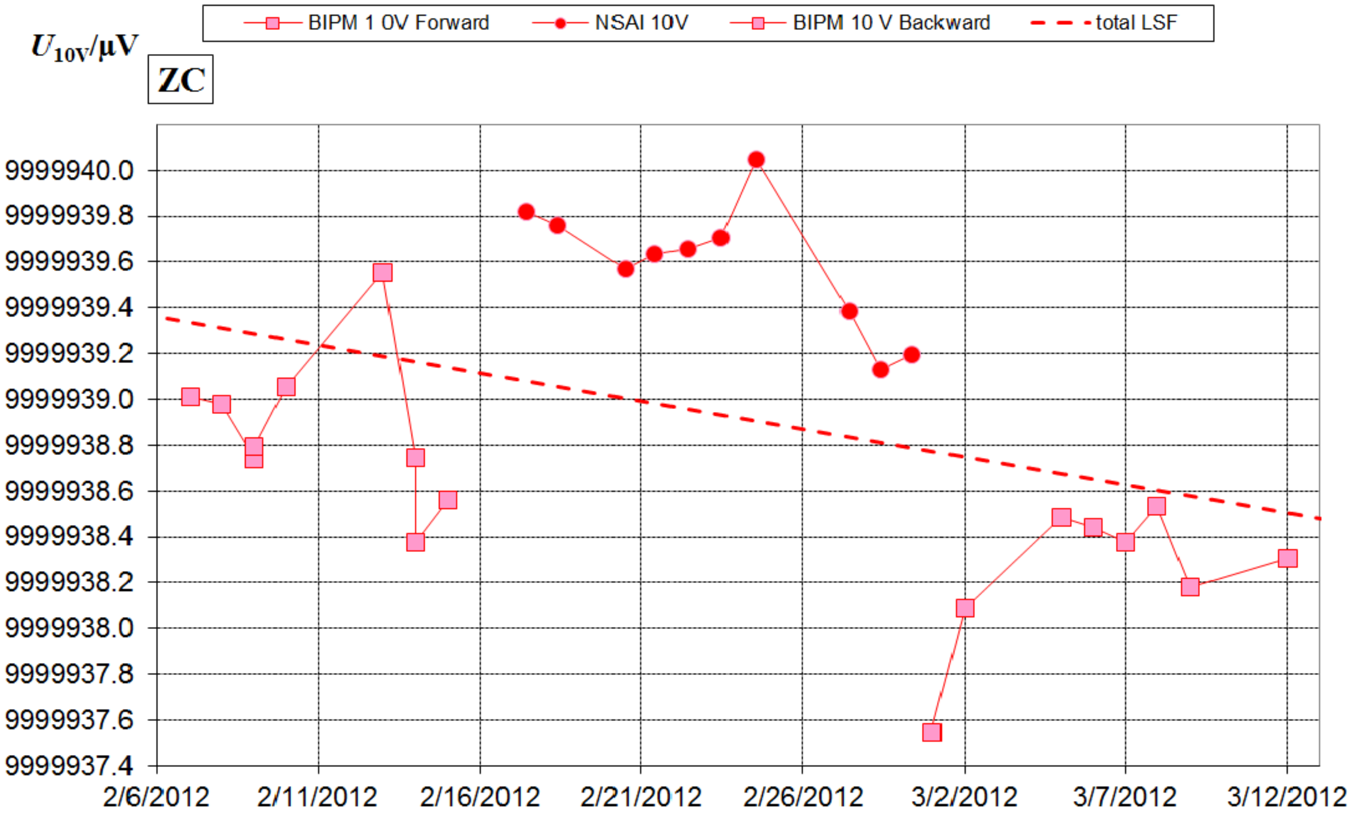

Figure 1a: Voltage of ZC at 10 V measured at both institutes (red squares for BIPM and red disks for NSAI) as a function of time, with a linear least-squares fit adjustment.

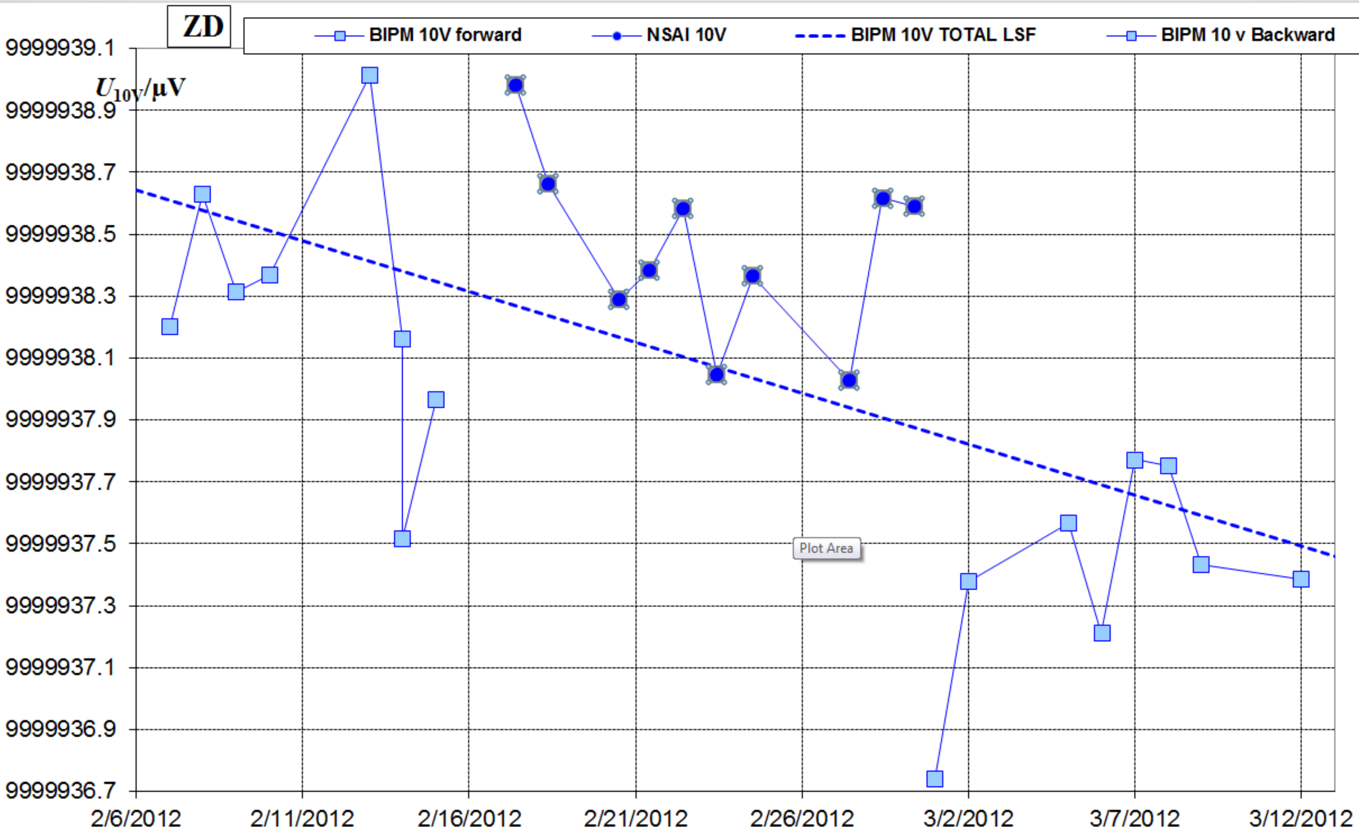

Figure 1b: Voltage of ZD at 10 V measured at both institutes (blue squares for BIPM and blue disks for NSAI) as a function of time, with a linear least-squares fit adjustment.

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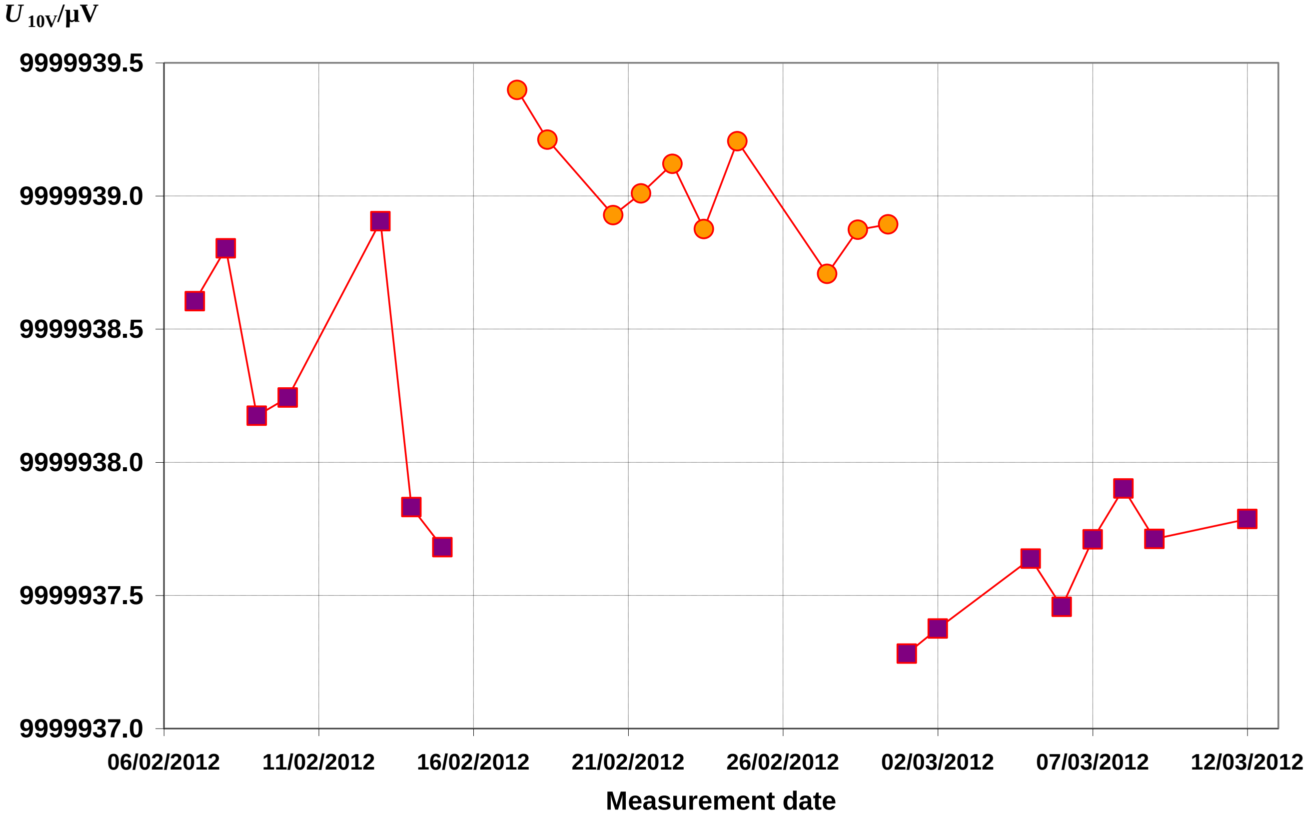

Figure 2: Voltage evolution of the simple mean of the two standards at 10 V.
NML measurements are represented by circles and BIPM measurements by squares.

Table 1 lists the results of the comparison and the uncertainty contributions for the comparison NSAI-NML/BIPM at 10 V. The relative value of the voltage noise floor due to flicker noise is about 1 part in 10^8 and represents the ultimate limit of the stability of Zener voltage standards.

Table 1. Results of the NSAI-NML (Ireland)/BIPM bilateral comparison of 10 V standards using two Zener traveling standards: reference date 23 February 2012. Uncertainties are 1 σ estimates.

		BIPM_D	BIPM_C	
1	*NSAI-NML (Ireland)* $(U_Z - 10\ \text{V})/\mu\text{V}$	-61.55	-60.41	
2	Type A uncertainty/µV	0.5	0.5	r
3	correlated unc. /µV	1.3		s
4	*BIPM* $(U_Z - 10\ \text{V})/\mu\text{V}$	-62.16	-61.45	
5	Type A uncertainty/µV	0.1	0.1	t
6	correlated unc./µV	0.001		u
7	pressure and temperature correction uncertainty/µV	0.03	0.02	v
8	$(U_{\text{NML}} - U_{\text{BIPM}})/\mu\text{V}$	0.62	1.04	
9	uncorrelated uncertainty/µV	0.51	0.51	w
10	$< U_{\text{NML}} - U_{\text{BIPM}} >/\mu\text{V}$	+0.83		
11	*a priori* uncertainty/µV	0.36		x
12	*a posteriori* uncertainty/µV	0.21		
13	correlated uncertainty/µV	1.3		y
14	comparison total uncertainty/µV	1.35		

The uncorrelated uncertainty is $w = [r^2 + t^2 + v^2]^{1/2}$.

The correlated uncertainty is $y = [s^2 + u^2]^{1/2}$.

As the *a priori* uncertainty and the *a posteriori* uncertainty are significantly different, we consider the largest component (*a priori* uncertainty $x = ½\,[w_A{}^2 + w_C{}^2\,]^{1/2}$) as the transfer uncertainty.

r is the NML Type A uncertainty (2);
s is the NML Type B uncertainty, which is assumed to be correlated for both transfer standards (3);
t is the BIPM Type A uncertainty (5);
u is the BIPM Type B uncertainty, which is assumed to be correlated for both transfer standards (6);
v is the pressure and temperature coefficient correction uncertainty (7);
w_i is the quadratic combination of the uncorrelated uncertainties for the Zener (9);
x is the uncertainty of the mean based on internal consistency (11);
y is the quadratic combination of the correlated uncertainties (13).

In estimating the uncertainty related to the stability of the standards during transportation, we have calculated the "*a priori"* uncertainty of the mean of the results obtained for the two standards (also called statistical internal consistency). It consists of the quadratic combination of the uncorrelated uncertainties of each result. We compared this component to the "*a posteriori"* uncertainty (also called statistical external consistency) which consists of the experimental standard deviation of the mean of the results from the two traveling standards*. If the "*a posteriori"* uncertainty is significantly larger than the "*a priori"* uncertainty, we assume that a standard has changed in an unusual way and we use the larger of these two estimates in calculating the final uncertainty.

In Table 1, the following elements are listed:

(1) the value attributed by NSAI-NML to each Zener U_{NML} , computed as the simple mean of all data from NSAI-NML;
(2) the Type A uncertainty which is the experimental standard deviation of the measurements performed at NSAI-NML;
(3) the uncertainty component arising from the maintenance of the volt at NSAI-NML: this uncertainty is completely correlated between the different Zeners used for a comparison;
(4-6) the corresponding quantities for the BIPM referenced to the mean date of NSAI-NML's measurements;
(7) the uncertainty due to the combined effects of the pressure and temperature coefficients and of the differences of the mean pressures and temperatures in the participating laboratories is calculated using the following assumption:
The uncertainty on the temperature correction is determined for the difference between the mean values of the temperature measured at both institutes which is then multiplied by the uncertainties of the temperature coefficients of each Zener standard.
$u_{\mathrm{T\text{-}i}} = U \times u(c_{\mathrm{T\text{-}i}}) \times \Delta R_i$ where $U = 10$ V, $u(c_{\mathrm{T\text{-}ZD}}) = 0.84\times10^{-7}$ /kΩ, $u(c_{\mathrm{T\text{-}ZC}}) = 0.48\times10^{-7}$ /kΩ and $\Delta R = 0.02$ kΩ for ZD and $\Delta R = 0.02$ kΩ for ZC.
The same procedure is applied for the uncertainty on the pressure correction for the difference between the mean values of the pressure measured at both institutes:
$u_{\mathrm{P\text{-}i}} = U \times u(c_{\mathrm{P\text{-}i}}) \times \Delta P_i$ where $U = 10$ V, $u(c_{\mathrm{P\text{-}ZD}}) = 0.096\times10^{-9}$ /hPa, $u(c_{\mathrm{P\text{-}ZC}}) = 0.056\times10^{-9}$ /hPa, $\Delta P = 6$ hPa for ZD and $\Delta P = 6$ hPa for ZC.
Note: the uncertainty on the measurement of the temperature and the pressure are negligible.

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[*] With only two traveling standards, the uncertainty of the standard deviation of the mean is comparable to the value of the standard deviation of the mean itself.

(8) the difference ($U_{\mathrm{NML}} - U_{\mathrm{BIPM}}$) for each Zener, and (9) the uncorrelated part of the uncertainty;

(10) the result of the comparison is the simple mean of the differences of the calibration results for the different standards;

(11 and 12) the uncertainty related to the transfer, estimated by the following two methods:

(11) the *a priori* uncertainty, determined as described on page 3;

(12) the *a posteriori* uncertainty, which is the standard deviation of the mean of the two results;

(13) the correlated part of the uncertainty and

(14) the total uncertainty of the comparison, which is the root sum square of the correlated part of the uncertainty and of (11) .

The comparison result is presented as the difference between the value assigned to a 10 V standard by NSAI-NML, at NSAI-NML, U_{NML}, and that assigned by the BIPM, at the BIPM, U_{BIPM}, which for the reference date is

$$\boldsymbol{U_{\mathrm{NML}} - U_{\mathrm{BIPM}} = +\ 0.83\ \mu\mathrm{V}; \quad u_{\mathrm{c}} = 1.35\ \mu\mathrm{V} \quad \text{on 2012/02/23}},$$

where u_{c} is the combined standard uncertainty associated with the measured difference, including the uncertainty of the representation of the volt at NSAI-NML, at the BIPM (based on $K_{\mathrm{J-90}}$), and the uncertainty related to the comparison.

Table 2 summarizes the uncertainties related to the calibration of a Zener diode against the Josephson array voltage standard at the BIPM.

Table 3 lists the uncertainties related to the calibration of a Zener at the NSAI-NML. Note that the uncertainty of the temperature (3) and pressure (4) corrections are given as an indication and do not appear in the final uncertainty budget as they are already contained in the comparison uncertainty budget.

Uncertainty Budgets

Table 2. Estimated standard uncertainties for Zener calibrations with the BIPM equipment at the level of 10 V without the contribution of the Zener noise. The standard deviation of the mean of the BIPM daily measurement results is equal to 139 nV

JVS & detector uncertainty components	Uncertainty/nV
Residual thermal electromotive forces	included in the Type A uncertainty
Residual EMF of the reversing switch	0.86
detector gain	0.11
leakage resistance	3×10^{-2}
frequency	3×10^{-2}
pressure and temperature correction	included in the Zener unc. budget
total	0.87

Note: We consider that the Type A uncertainty can't be lower than the 1/f noise floor estimated at 100 nV.

Table 3. Estimated standard uncertainties for Zener calibrations with the NSAI-NML equipment at the level of 10 V.

The measurement model is: $U_X = U_{REF} \cdot + f(\delta U_i) + \delta p + \delta T$

Input Quantity	**Symbol**	**Standard Uncertainty**	**Sensitivity Coefficient**	**Uncertainty Contribution**	**Note**
		(μV)		(μV)	
NMLI Reference	U_{REF}	1.2	1	1.2	(1)
Voltage difference	$f(\delta U_i)$	0.5	1	0.5	(2)
Temperature correction	δ_T	0.01	1	0.01	(3)
Pressure Correction	δ_P	0.03	1	0.04	(4)
Non-repeatability		0.5	1	0.5	(5)
		Combined Standard Uncertainty		1.39	
		Expanded Uncertainty (*k*=2)		2.8	

Notes:

(1) The uncertainty component includes the effects of drift, noise, and environmental influences on the ensemble reference standard.
(2) The uncertainty component includes the effects of uncompensated thermal voltage offsets, uncorrected errors in the detector reading, leakage effects, and common mode effects.
(3) A temperature coefficient of 3.5×10^{-7} kΩ^{-1} is used.
(4) A pressure coefficient of 2×10^{-8} kPa^{-1} is used.
(5) An estimate of the 1/f noise floor level is used as it is greater than the standard deviation of the mean.

Consistency of the results with the behavior of the standards at BIPM.

As the difference between the results of NSAI-NML and BIPM is larger than those obtained for the same exercise in the recent years, we have checked the behavior of the two transfer standards for a possible non consistency using the latest calibration results of the BIPM:

The results obtained during the bilateral comparison have been inserted into the graph presenting the calibration history of the standards at BIPM (Fig 3, Fig. 4a and 4b).

The “check” measurements are carried out to establish the long term behavior of the BIPM secondary standards: each Zener is calibrated against a reference value which consists of the simple mean of the output voltage of two very reliable secondary standards (Zeners-723A Type). These two standards are directly calibrated against the JVS. These “check” measurements are simpler than direct calibrations against the Josephson arrays and can be carried out more often.

The results obtained using this technique are represented by green diamonds on Fig. 4a and 4b. The results of the “check” measurements and the calibrations against the Josephson array agree very well for BIPM_C and BIPM_D, even if the two techniques are strongly correlated by their link to the primary standard.

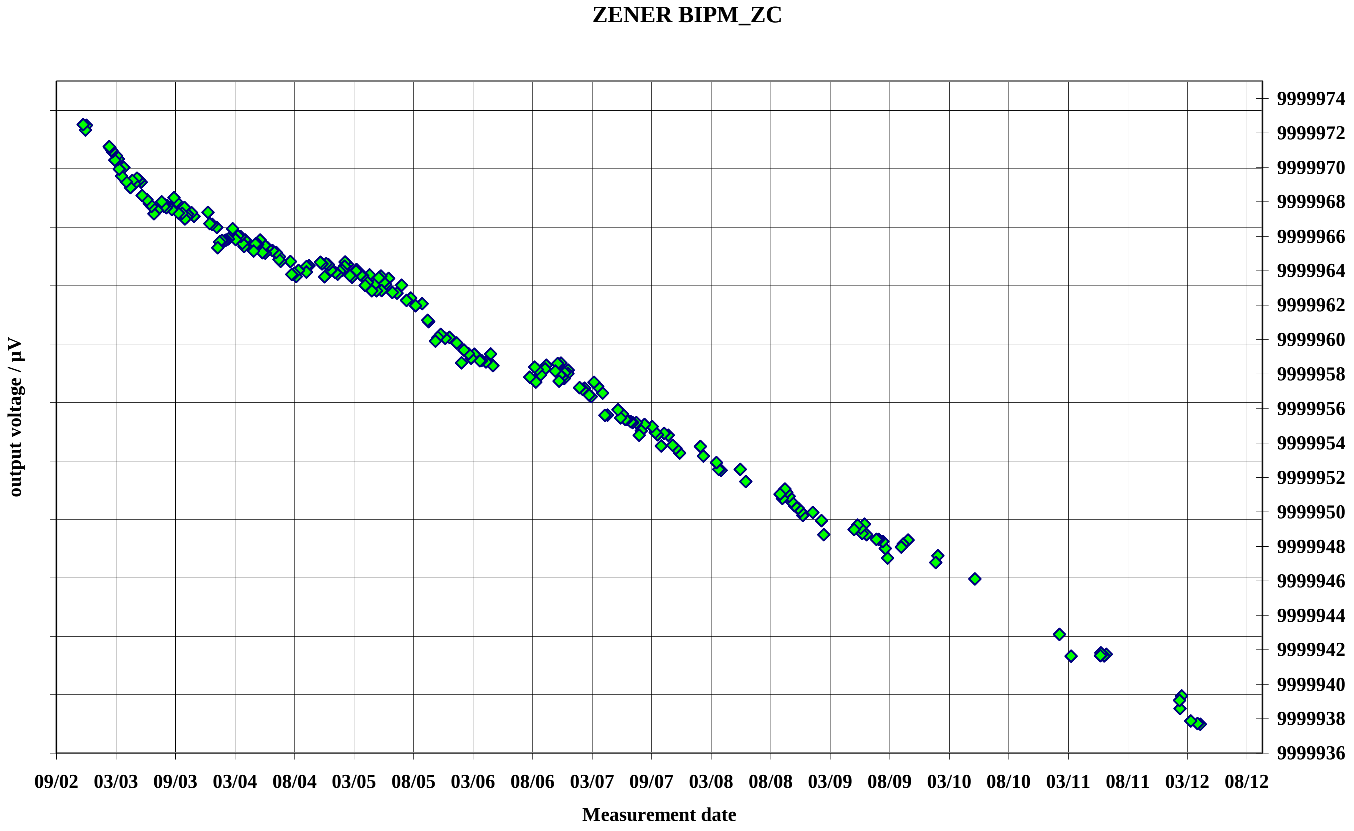

Figure 3: example of the voltage evolution of BIPM_C, established using the simple “check” technique at BIPM over the period from September 2002 to March 2012 (see the text for details).

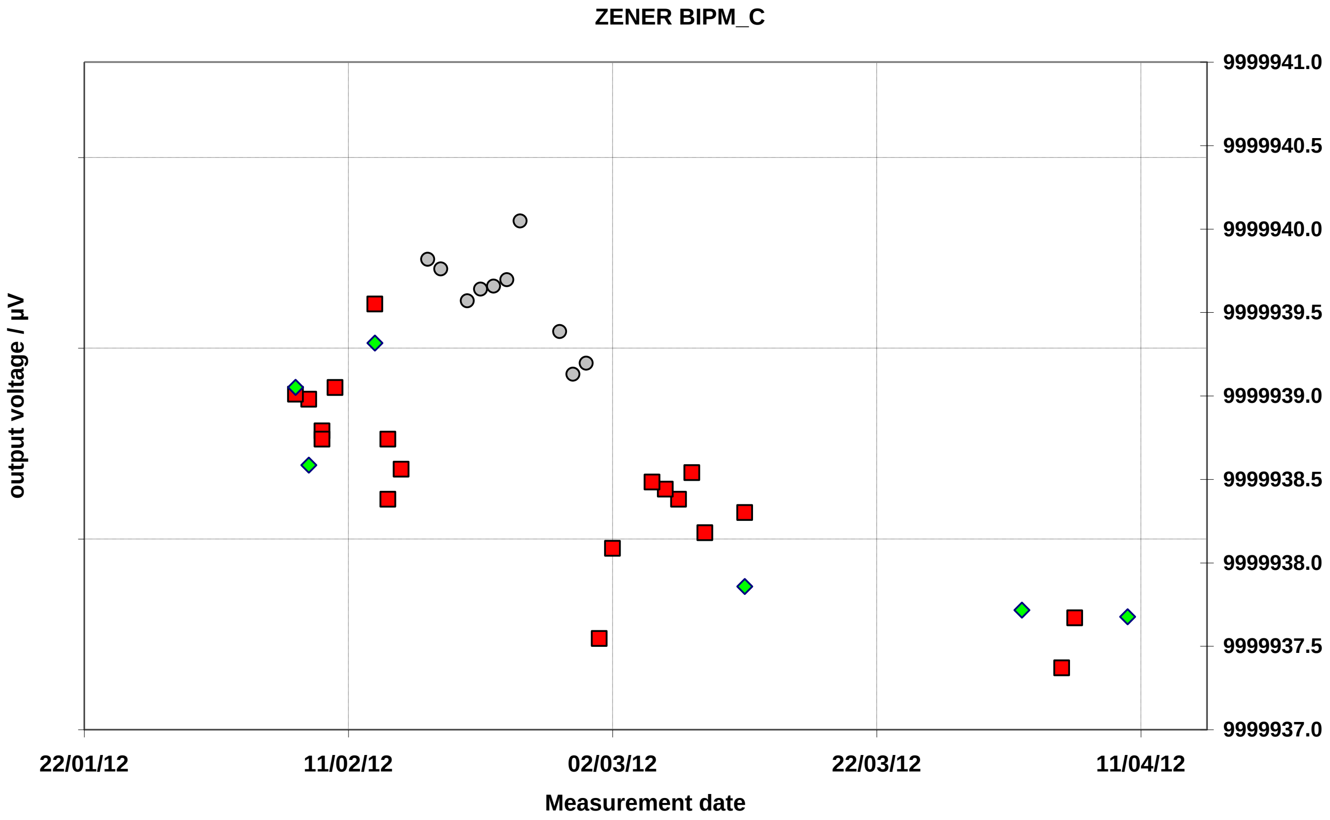

Figure 4a: Voltage evolution of BIPM_C: The red squares are the BIPM measurements for the bilateral comparison. The grey disks are the NSAI measurements for the same exercise and the green diamonds are the BIPM "check" measurements.

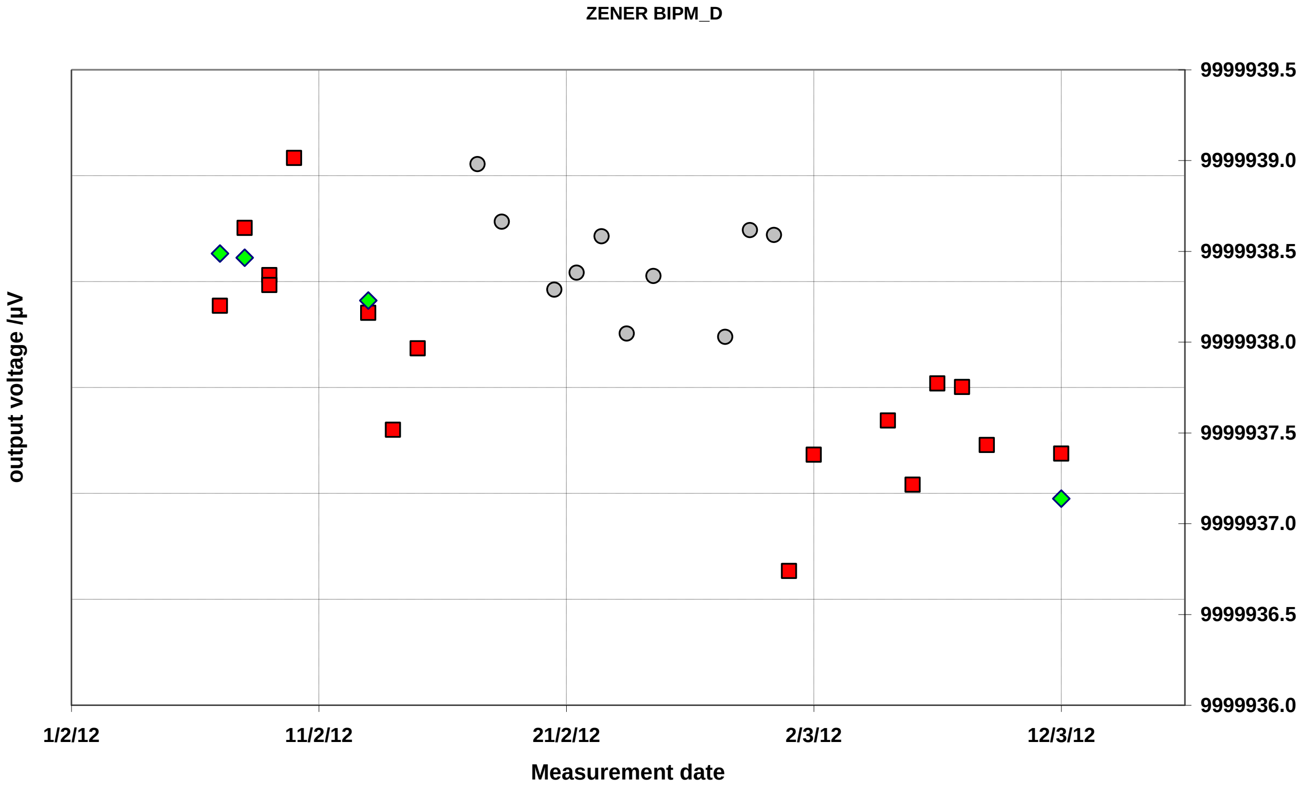

Figure 4b: Voltage evolution of BIPM_D: The red squares are the BIPM measurements for the bilateral comparison. The grey disks are the NSAI measurement for the same exercise and the green diamonds are the BIPM "check" measurements.

Evaluation of the pressure correction coefficients

The pressure correction coefficients of the two transfer standards haven't been checked since 2004 and it can be assumed that they have changed over 8 years.

In 2004, the coefficients were determined using a dedicated measurement system where the pressure of a chamber containing the Zener could be varied. The corresponding change in the voltage output was recorded [1].

As BIPM Zeners standards are monitored several times during the year, we have collected a large number of voltage output values together with the corresponding atmospheric pressure value at the time of the measurement. Assuming a "natural" linear drift of the voltage output and a correlated atmospheric pressure variation, a least squares fit adjustment can be applied to the data and the corresponding pressure correction coefficient can be determined

For each standard we have applied this method for 140 measurements covering the period from March 2005 to May 2012. Table 4 presents the results of the least square adjustment.

	ZC	ZD
Pressure coefficient 10^9/hPa measured in 2004	1.90	1.83
Pressure coefficient 10^9/hPa calculated in 2012	2.03	3.21
Determination coefficient of the 2012 determination	0.9925	0.9929

If we apply these "new" pressure correction coefficients to the comparison data and re-calculate the final result, we end with a voltage difference $U_{\mathrm{NML}} - U_{\mathrm{BIPM}} = + 0.87\ \mu\mathrm{V}$, that is to say a shift of 40 nV of the final comparison result. This change is not significant in regards with the most important uncertainty component and confirms the calculated final result.

As it has been demonstrated [2], the difference between the two determined values of the pressure correction coefficient is due to an irregular drift behaviour of the standards which could not be fully compensated for.

Conclusion

The final result of the comparison is presented as the difference between the value assigned to DC voltage standard by NSAI-NML, at the level of 10 V, at NSAI-NML, U_{NML}, and that assigned by the BIPM, at the BIPM, U_{BIPM}, at the reference dates of the 23rd of February 2012.

$$\boldsymbol{U_{\mathrm{NML}} - U_{\mathrm{BIPM}} = +\,0.83\ \mu\mathrm{V}, \quad u_{\mathrm{c}} = 1.35\ \mu\mathrm{V}, \quad \text{at 10 V}}$$

where u_{c} is the combined standard uncertainty associated with the measured difference, including the uncertainty of the representation of the volt at the BIPM and at NSAI-NML, based on $K_{\mathrm{J-90}}$, and the uncertainty related to the comparison.

The final result is impacted by the anomalous offset between the NSAI-NML results for the two transfer standards (Cf. Fig.1). The reason for this offset hasn't been determined.

However the difference remains within the total combined standard uncertainty. Therefore, the comparison result shows that the voltage standards maintained by NSAI-NML and the BIPM were equivalent, within their stated expanded uncertainties, on the mean date of the comparison.

References

[1] Witt T.J., *Maintenance and dissemination of voltage standards by Zener-diode-based instruments*, IEE Proceedings 6(149), p305-312, Nov. 2002

[2] O. Power and J. E. Walsh, In-service characterization of electronic voltage standards, *IEEE Trans. Instrum. Meas.*, 54(2):559-562, Apr 2005