

**Bilateral Comparison of 10 V Standards  
between the NSAI - NML (Ireland) and the BIPM,  
March 2014  
(part of the ongoing BIPM key comparison BIPM.EM-K11.b)**

by S. Solve<sup>+</sup>, R. Chayramy<sup>+</sup>, O. Power\* and M. Stock<sup>+</sup>

<sup>+</sup> Bureau International des Poids et Mesures, Sèvres, France

\* National Standards Authority of Ireland - National Metrology Laboratory  
(NSAI - NML), Dublin, Ireland

## **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 in February and March 2014. Two BIPM Zener diode-based travelling standards (Fluke 732B), BIPM\_4 (Z4) and BIPM\_5 (Z5), 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 of the Zener standards on internal temperature and ambient atmospheric pressure.

## **Outline of the measuring method**

### **NSAI-NML 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 1  $\mu\text{V}$ , the nanovoltmeter is set to its 10  $\mu\text{V}$  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 inverting 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 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. If this occurs, the "Data Acquisition" stage starts again. 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

Figure 1 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 (2014/03/10).

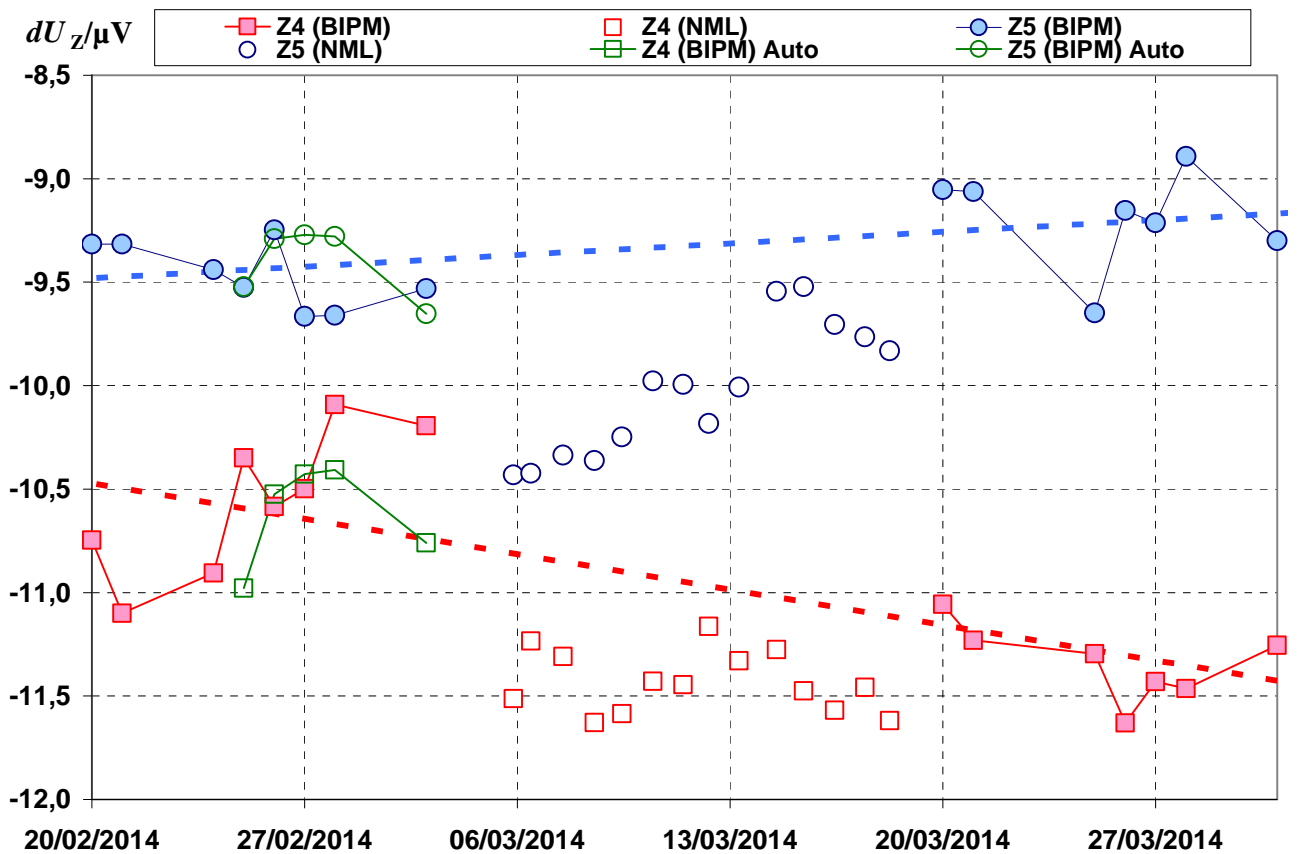


Figure 1: Voltage of Z4 (red squares) and Z5 (blue disks) at 10 V measured at both institutes, referred to an arbitrary origin, as a function of time, with a linear least-squares fit to the measurements of BIPM. The green circles and squares are BIPM measurements carried out with an independent automated measurement setup based on a primary standard (cf. Appendix A)

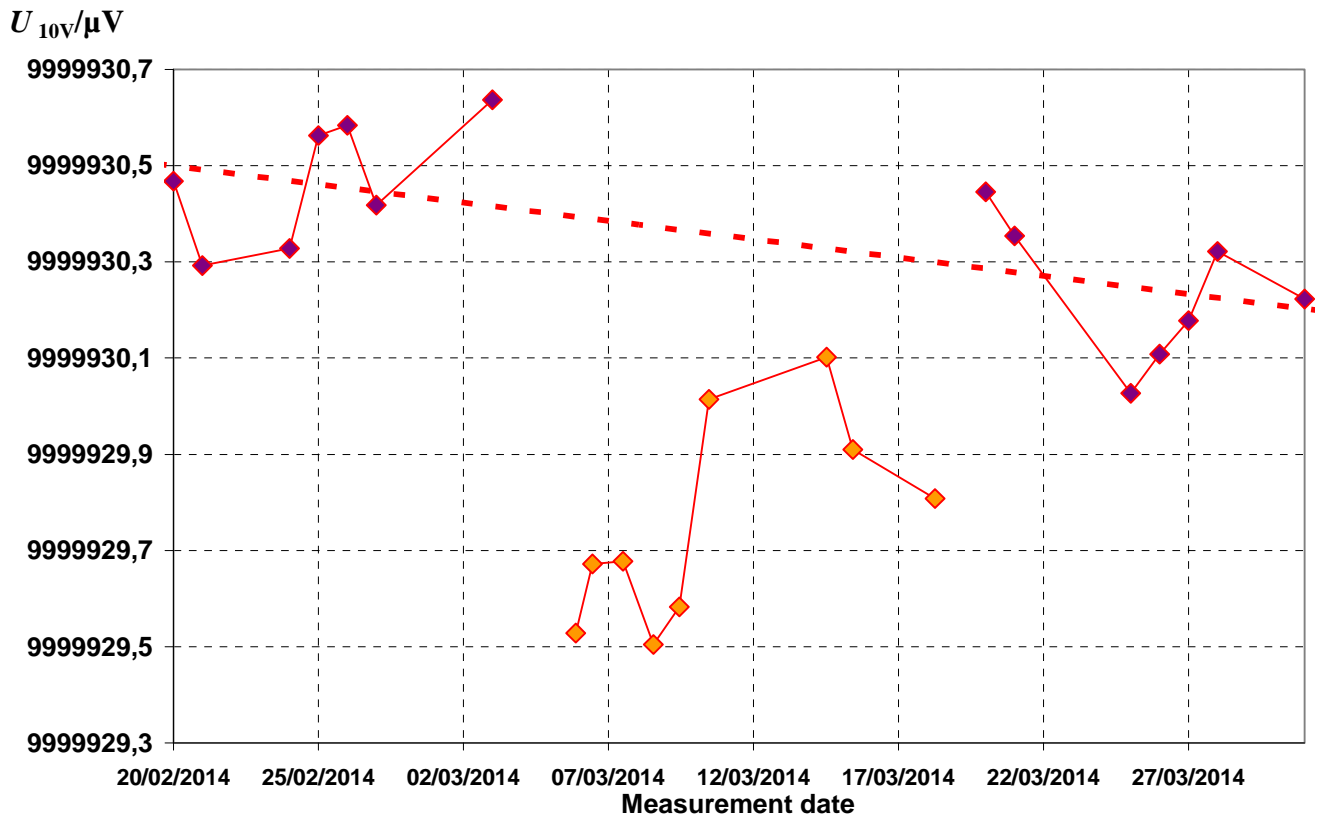


Figure 2: Voltage evolution of the simple mean of the two standards at 10 V.

NML measurements are represented by yellow diamonds and BIPM measurements by violet diamonds.

Table 1 lists the results of the comparison and the uncertainty contributions for the comparison NSAI-NML/BIPM at 10 V. The estimated relative value of the voltage noise floor due to flicker noise is about 1 part in  $10^8$  at BIPM (while 5 parts in  $10^8$  at NML) and represents the ultimate limit of the stability of Zener voltage standards.

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.

\* 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.

In Table 1, the following elements are listed:

- (1) the value attributed by NSAI-NML to each Zener  $U_{\text{NML}}$ , computed as the simple mean of all data from NSAI-NML;
- (2) the NSAI-NML estimated Type A uncertainty. The experimental standard deviation of the mean of the measurements performed at NSAI-NML are 0.079  $\mu\text{V}$  and 0.108  $\mu\text{V}$  for Z4 and Z5 respectively;
- (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. Exception made with the value attributed by BIPM to each Zener  $U_{\text{BIPM}}$ , is computed from a linear fit on all BIPM measurements;  $U_{\text{BIPM}}$  is 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 of the temperature correction  $u_{T,i}$  of Zener  $i$  is determined for the difference  $\Delta R_i$  between the mean values of the thermistor resistances measured at both institutes which is then multiplied by the uncertainties  $u(c_{T,i})$  of the temperature coefficients of each Zener standard:

$$u_{T,i} = U \times u(c_{T,i}) \times \Delta R_i$$

where  $U = 10 \text{ V}$ ,  $u(c_{T,Z4}) = 1.25 \times 10^{-7} / \text{k}\Omega$ ,  $u(c_{T,Z5}) = 1.55 \times 10^{-7} / \text{k}\Omega$  and  $\Delta R_{Z4} = 0.011 \text{ k}\Omega$  and  $\Delta R_{Z5} = 0.081 \text{ k}\Omega$ .

The same procedure is applied for the uncertainty  $u_{P,i}$  on the pressure correction for the difference  $\Delta P_i$  between the mean values of the pressure measured at both institutes:

$$u_{P,i} = U \times u(c_{P,i}) \times \Delta P_i$$

where  $U = 10 \text{ V}$ ,  $u(c_{P,Z4}) = 0.11 \times 10^{-9} / \text{hPa}$ ,  $u(c_{P,Z5}) = 0.06 \times 10^{-9} / \text{hPa}$ ,  $\Delta P_{Z4} = \Delta P_{Z5} = 18.2 \text{ hPa}$ .

Note: the uncertainty on the measurement of the temperature and the pressure are negligible.

- (8) the difference ( $U_{\text{NML}} - U_{\text{BIPM}}$ ) 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 5;
  - (12) the *a posteriori* uncertainty, also described on page 5;

(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).

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 included separately in the comparison uncertainty budget (Table 1).

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

$$U_{\text{NML}} - U_{\text{BIPM}} = -0.64 \mu\text{V}; \quad u_c = 1.35 \mu\text{V} \quad \text{on 2014/03/10,}$$

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  $K_{\text{J-90}}$ ), and the uncertainty related to the comparison.

**Table 1. Results of the NSAI-NML (Ireland)/BIPM bilateral comparison of 10 V standards using two Zener traveling standards: reference date 10 March 2014. Uncertainties are 1  $\sigma$  estimates.**

		BIPM_4	BIPM_5	
1	NSAI-NML (Ireland) ( $U_z - 10\text{ V}$ )/ $\mu\text{V}$	-66.43	-74.15	
2	Type A uncertainty/ $\mu\text{V}$	0.50	0.50	<i>r</i>
3	correlated unc./ $\mu\text{V}$	1.30		<i>s</i>
4	BIPM ( $U_z - 10\text{ V}$ )/ $\mu\text{V}$	-65.96	-73.33	
5	Type A uncertainty/ $\mu\text{V}$	0.10	0.10	<i>t</i>
6	correlated unc./ $\mu\text{V}$	0.01		<i>u</i>
7	pressure and temperature correction uncertainty/ $\mu\text{V}$	0.02	0.13	<i>v</i>
8	( $U_{\text{NML}} - U_{\text{BIPM}}$ )/ $\mu\text{V}$	-0.47	-0.82	
9	uncorrelated uncertainty/ $\mu\text{V}$	0.51	0.53	<i>w</i>
10	$\langle U_{\text{NML}} - U_{\text{BIPM}} \rangle$ / $\mu\text{V}$	-0.64		
11	<i>a priori</i> uncertainty/ $\mu\text{V}$	0.37		<i>x</i>
12	<i>a posteriori</i> uncertainty/ $\mu\text{V}$	0.17		
13	correlated uncertainty/ $\mu\text{V}$	1.30		<i>y</i>
14	comparison total uncertainty/ $\mu\text{V}$	1.35		

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

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



## Uncertainty Budgets

**Table 2.** The following table presents the estimated standard uncertainties arising from the JVS and the measurement setup for Zener calibrations with the BIPM equipment at the level of 10 V without the contribution of the Zener noise.

<b>JVS &amp; detector uncertainty components</b>	<b>Uncertainty/nV</b>
Noise of the measurement loop that includes the residual thermal electromotive forces including the residual EMF of the reversing switch	0.86
detector gain	0.11
leakage resistance	$3 \times 10^{-2}$
frequency	$3 \times 10^{-2}$
pressure and temperature correction	included in the Zener uncertainty budget
total	0.87

*Note: The standard deviation of the mean of the BIPM daily measurement results is equal to 48 nV. However, 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:  $U_X = U_{REF} \cdot f(\delta U_i) + \delta\rho + \delta T$

Input Quantity	Symbol	Standard Uncertainty	Sensitivity Coefficient	Uncertainty Contribution	Note
		( $\mu\text{V}$ )		( $\mu\text{V}$ )	
NSAI-NML Reference	$U_{REF}$	1.2	1	1.2	(1)
Voltage difference	$f(\delta U_i)$	0.5	1	0.5	(2)
Temperature correction	$\delta_T$	0.01	1	0.01	(3)
Pressure Correction	$\delta_P$	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  $3.5 \times 10^{-7} \text{ k}\Omega^{-1}$  is used.
- (4) A pressure coefficient of  $2 \times 10^{-8} \text{ kPa}^{-1}$  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.

## 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,  $U_{\text{NML}}$ , and that assigned by the BIPM, at the BIPM,  $U_{\text{BIPM}}$ , at the reference dates of the 10<sup>th</sup> of March 2014.

$$U_{\text{NML}} - U_{\text{BIPM}} = - 0.64 \mu\text{V}, \quad u_c = 1.35 \mu\text{V}, \quad \text{at } 10 \text{ V}$$

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  $K_{\text{J-90}}$ , and the uncertainty related to the comparison.

This is a satisfactory result. The comparison result shows that the voltage standards maintained by NSAI-NML and the BIPM were equivalent, within their stated standard uncertainties, on the mean date of the comparison.

## Appendix A: Upgrade of the BIPM automated Zeners measurement setup

The BIPM automated measurement setup that was developed at the level of 1 V in the recent years is being upgraded to reach 10 V. The 1 V system was based on a PTB programmable array [1-2] made of SINIS (Superconductor/Isolator/Normal Metal/ Isolator/ Superconductor) Josephson junctions and is being replaced with a 10 V PTB programmable array based on the new SNS (Superconductor/Normal Metal/ Superconductor) junctions technology [3].

We took the opportunity of this comparison exercise to perform in parallel of the official measurements of the transfer standards with our SIS based-measurement setup some measurements with our automated measurement setup upgraded to the 10 V level.

The results are presented on Figure 1 and in the following Table A1:

	Z4 / $\mu\text{V}$	Z5 / $\mu\text{V}$
Mean value (5 meas. days) -SIS	9999934,66	9999926,47
Std. Dev. of the mean - SIS	0,092	0,076
Mean value (5 meas. days) -SNS	9999934,38	9999926,60
Std. Dev. of the mean - SNS	0,110	0,078
“SNS” – “SIS”	-0,28	0,13

Table 1: Comparison of the measurements of the transfer standards carried out with 2 fully independent measurement setups at 10 V in parallel over 5 consecutive days.

[1] Solve S., Chayramy R., Reymann D., A new fully automated measurement chain for electronic voltage standards at 1.018 V, *IEEE Trans. Instrum. Meas.*, 2007, 56, 588-591

[2] Solve S., Chayramy R., Stock M., The BIPM 1.018 V Zener Measurement Set-up, *Rapport BIPM-2011/05*, 32 pp.

[3] Mueller F., Behr R., Weimann T., Palafox L., Olaya D., Dresselhaus P.D., Benz S.P., 1 V and 10 V SNS Programmable Voltage Standards for 70 GHz, *IEEE Trans. Appl. Super.*19(3), 981-86, June 2009.