Bilateral Comparison of 1 V and 10 V Standards
between the NIS (Egypt) and the BIPM,
August to September 2014
(part of the ongoing BIPM key comparison BIPM.EM-K11.a and b)

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Introduction

As part of the ongoing BIPM key comparison BIPM.EM-K11.a and b, a comparison of the 1 V and 10 V voltage reference standards of the BIPM and the National Institute for Standards (NIS)**,** Giza, Egypt, was carried out from August to September 2014. Two BIPM Zener diode-based travelling standards (Fluke 732B), BIPM_B (ZB) and BIPM_C (ZC), were transported as hand luggage on board an airplane to NIS and back to BIPM. At NIS, the reference standard for DC voltage is a Josephson Voltage Standard. The output EMF (Electromotive Force) of each travelling standard was measured by direct comparison with the primary standard.

At the BIPM, the travelling standards were calibrated, before and after the measurements at NIS, 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

NIS 1 V and 10 V measurements

The NIS system used in this comparison is a fully automated 10 Volt SIS Josephson Voltage Standard (JVS) called supraVOLT-control and manufactured by Supracon AG – Germany [1-2]. It is a complete 3-channel microprocessor-controlled JVS with a highly integrated low-Tc Josephson Junction (JJ) array microwave circuit. The JJ array is connected in series to a high resolution null detector (Keithley nanovoltmeter). Its self-calibration has been done before starting in the measurements. The nanovoltmeter measures the difference voltage between the Zener standard which has to be measured and the quantized voltage level of the JJ array chip for both polarities. With these two readings the software calculates the voltage of the Zener standard by eliminating the thermal voltage. Twenty measurements in both polarities have been made in order to determine the difference voltage with a high accuracy. The polarity of the Josephson voltage is reversed by the polarity change of the bias current through the JJ array chip and the polarity of the Zener is reversed by the polarity reversal switch. During the measurement of the difference voltage, the JVS electronics is completely disconnected from the JJ array chip in order to avoid grounding problems. Each measured data point and its standard deviation is calculated from forty measurements (twenty in the positive and twenty in the negative polarity). The JJ array chip is based on standard Nb/Al trilayer technology.

At an operating frequency of 75 GHz the Josephon voltage standard circuit (JVSC) generate about 130,000 discrete voltage levels in the range of −10 V to +10 V at intervals of about 155 µV. The EIP source locking microwave counter is used for the stabilization of the microwave frequency of the 75 GHz Gunn oscillator. The frequency down converted output signal of the remote sensor of the microwave electronics is fed to the input of the EIP counter. This signal, and the 75 GHz signal of the Gunn oscillator, are phase locked to a 10 MHz external reference frequency. Thus, the accuracy and stability of 75 GHz microwave oscillator is the same as that of the 10 MHz reference frequency. The locking frequency of about 75 GHz can be adjusted in 10 kHz steps.

supraVOLT-control system includes sensors for the barometric pressure and internal temperature, which are integrated in the JVS electronics unit, and for humidity and environmental temperature. The test current of measuring the two terminal thermistor resistance which indicates the Zener standard temperature is adjusted at 10 µA to avoid heating of the thermistor.

The “GUARD” and “CHASSIS” binding post terminals of the Zener standard are connected together to a single point which is the grounding reference point of the measurement setup. The Zeners are disconnected from the mains at least two hours before the beginning of the measurements and they are reconnected to the mains at most six hours later.

BIPM Measurements for both 1 V and 10 V

The output voltage of the Zener standard to be measured is connected in series opposition to the BIPM Josephson Voltage Standard - Hypres 10 V SIS array (S/N: 2548E-6) - , through a low thermal Electromotive Forces (EMF) switch. The binding post terminals “GUARD” and “CHASSIS” of the Zener standard are connected together 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 in order for the Zener internal temperature to stabilize.

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.

The BIPM array biasing frequency has been adjusted to a value where the voltage difference between the primary and the secondary voltage standards is below 0.5 µV for both nominal voltages. The nanovoltmeter is set to its 3 µV range for the measurements performed at the level of 1 V and on its 10 µV range for those carried out at the level of 10 V. 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 internal linear thermo-electromotive forces and to check that there is no AC voltage noise rectified at the input of the detector (this is the case if the reading is different in the positive and negative polarity of the analog detector by a few hundreds of a microvolt).

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 or the software warns the operator with a beep. If too many beeps occur, the operator can reject the “Data Acquisition” sequence and start it again. The “Data Acquisition” sequence lasts 25 s and the array must remain on its quantum voltage step during this period of time. 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 which is used to compute the final result at 10 V.

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 NIS measurements (2014/09/07).

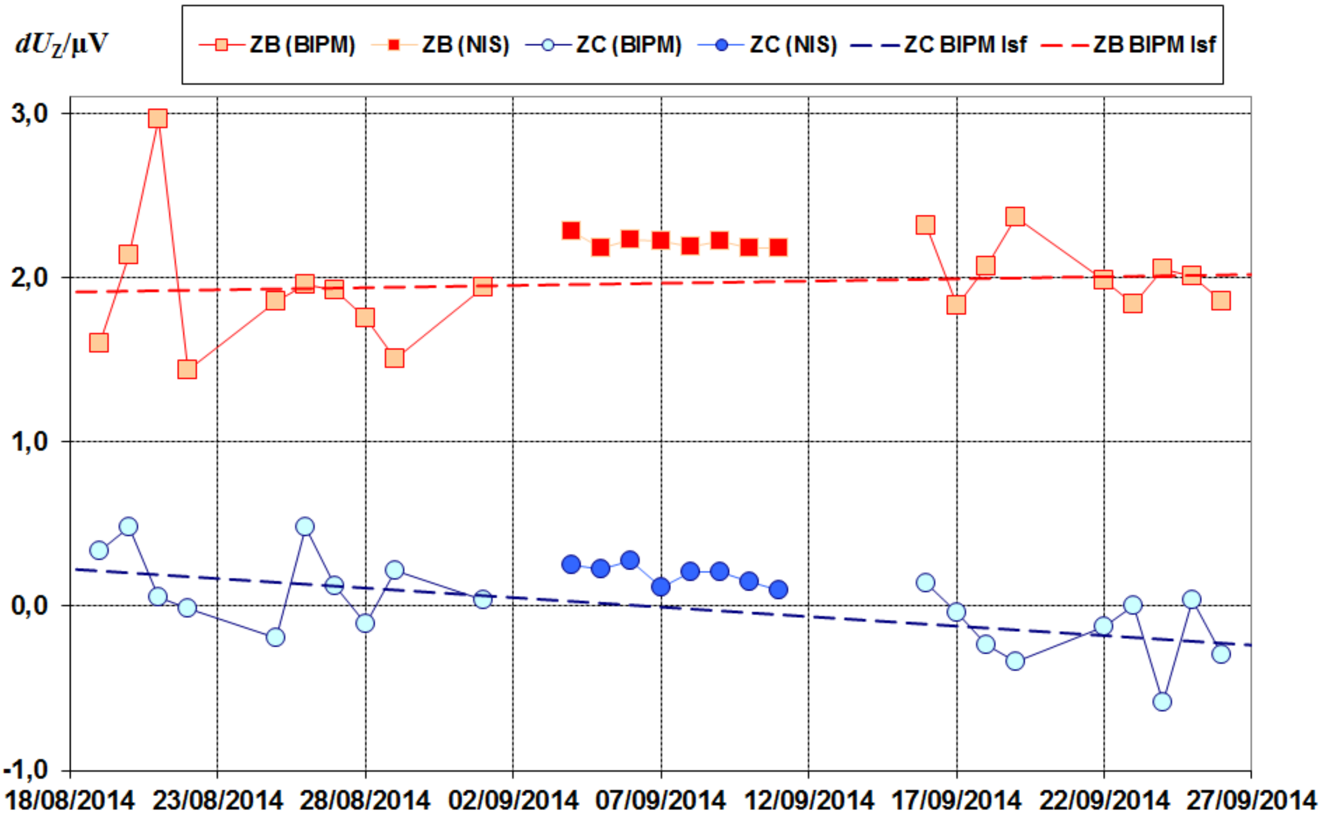

Figure 1a: Voltage of ZC (top) and ZB (bottom) at 10 V measured at both institutes (light markers for BIPM and dark markers for NIS), referred to an arbitrary origin as a function of time, with a linear least-squares fit (lsf) to the BIPM measurements.

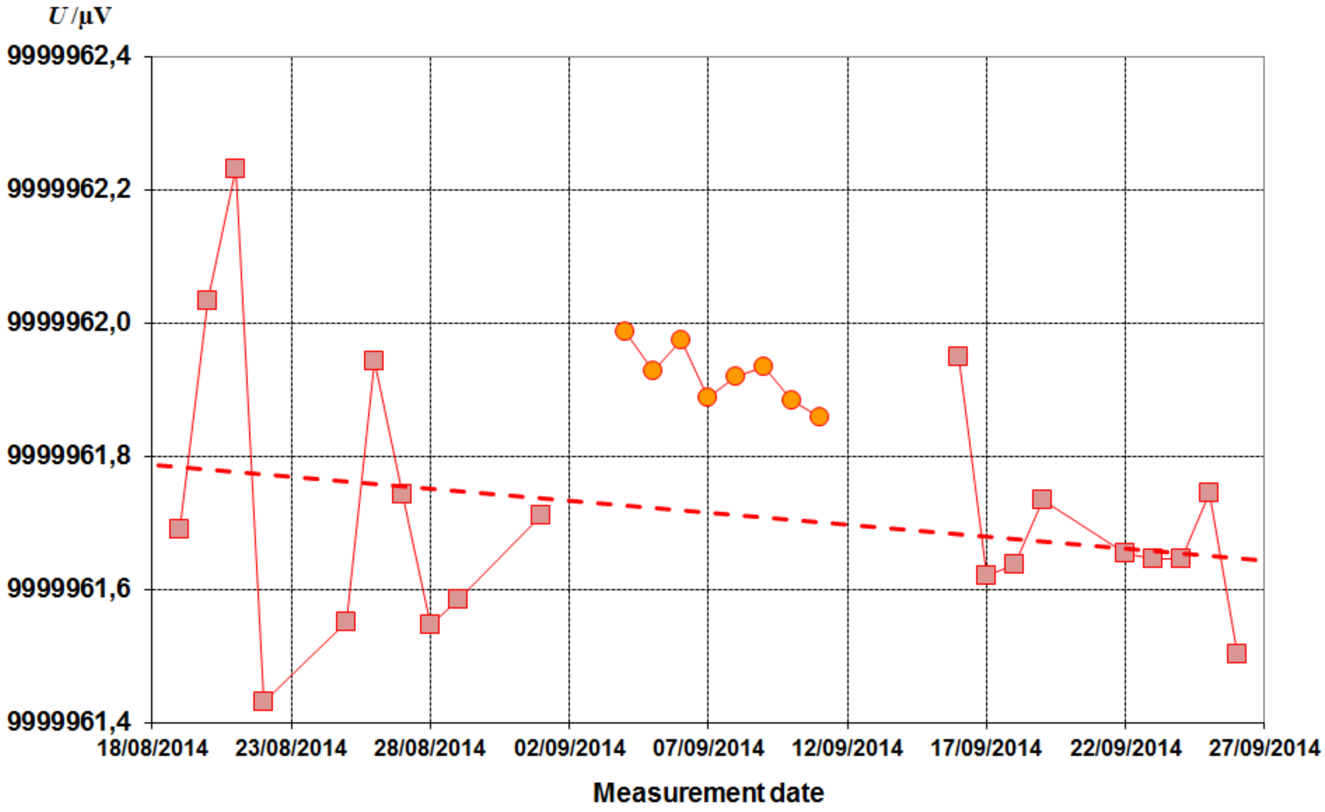

Figure 2: Voltage evolution of the simple mean of the two standards at 10 V.
NIS measurements are represented by disks and BIPM measurements by filled squares.

Table 1 lists the results of the comparison and the uncertainty contributions for the comparison NIS/BIPM at 10 V.

Table 1. Results of the NIS (Egypt)/BIPM bilateral comparison of 10 V standards using two Zener traveling standards: reference date 07 September 2014. Uncertainties are 1 σ estimates.

		BIPM_B	BIPM_C
1	*NIS (Egypt)* (U_Z – 10 V)/µV	-6.79	-69.37
2	Type A uncertainty/µV	0.1	0.1
3	correlated (Type B) unc. /µV	0.1	
4	*BIPM* (U_Z – 10 V)/µV	-7.03	-69.57
5	Type A uncertainty/µV	0.1	0.1
6	correlated (Type B) unc./µV	0.001	
7	pressure and temperature correction uncertainty/µV	0.003	0.008
8	(U_{NIS} – U_{BIPM})/µV	0.24	0.20
9	uncorrelated uncertainty/µV	0.142	0.142
10	< U_{NIS} – U_{BIPM} >/µV	0.219	
11	*a priori* uncertainty/µV	0.1	
12	*a posteriori* uncertainty/µV	0.021	
13	correlated uncertainty/µV	0.1	
14	comparison total uncertainty/µV	0.14	

In Table 1, the following elements are listed:

(1) the value attributed by NIS to each Zener U_{NIS}, computed as the simple mean of all data from NIS;

(2) the Type A uncertainty which is the larger of the experimental standard deviation of the mean of the measurements performed at NIS, and the 1/*f* noise floor of 100 nV which, according to the experience of the BIPM, in general limits the accuracy of Zener voltage standards [3].

(3) the uncertainty component arising from the maintenance of the volt at NIS: 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 NIS measurements;

(7) the uncertainty due to the combined effects of the uncertainties of the pressure and temperature coefficients[*] and to the differences of the mean pressures and temperatures in the participating laboratories is calculated using the following assumption:

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[*] The evaluation of the correction coefficients was performed in 2000.

The uncertainty on the temperature correction $u_{T,i}$ of Zener i is determined for the difference ΔR_i between the mean values of the thermistor resistances measured at both institutes which is then multiplied by the uncertainty $u(c_{T,i})$ of the temperature coefficient of this Zener standard:

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

where U = 10 V, $u(c_{\mathrm{T,ZB}}) = 0.63\times10^{-7}$ / kΩ, $u(c_{\mathrm{T,ZC}}) = 0.48\times10^{-7}$ / kΩ and $\Delta R_{ZB} = 0.005$ kΩ and $\Delta R_C = 0.016$ kΩ.

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

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

where U = 10 V, $u(c_{\mathrm{P,ZB}})= 0.0563\times10^{-9}$ / hPa, $u(c_{\mathrm{P\text{-}ZC}}) = 0.067\times10^{-9}$ / hPa, $\Delta P_{ZB} = 1.2$ hPa and $\Delta P_{ZC} = 0.95$ hPa.

The uncertainty on the measurement of the temperature is negligible.

(8) the difference ($U_{\mathrm{NIS}} - U_{\mathrm{BIPM}}$) for each Zener, and (9) the uncorrelated part of the uncertainty, calculated as the quadratic sum of lines 2, 5 and 7;

(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 the standard uncertainty of the mean, obtained by propagating the Type A uncertainties for both Zeners;
>
> (12) the *a posteriori* uncertainty, which is the standard deviation of the mean of the two results;

(13) the correlated part of the uncertainty, calculated as the quadratic sum of lines 3 and 6, and

(14) the total uncertainty of the comparison, which is the root sum square of the correlated part of the uncertainty and of the larger of (11) and (12).

To estimate 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

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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.

standard has changed in an unusual way, probably during their transportation, and we use the larger of these two estimates in calculating the final uncertainty.

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

$$\boldsymbol{U_{\mathrm{NIS}} - U_{\mathrm{BIPM}} = 0.22\ \mu\mathrm{V}; \qquad u_{\mathrm{c}} = 0.14\ \mu\mathrm{V} \quad \text{on 2014/09/07}},$$

where u_{c} is the combined standard uncertainty associated with the measured difference, including the uncertainty of the representation of the volt at NIS, at the BIPM (based on $K_{\mathrm{J\text{-}90}}$), 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 NIS. Note that the uncertainty of the temperature, pressure corrections and the contribution of the Zener noise (in italic) 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).

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.

JVS & detector uncertainty components	Uncertainty/nV
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	3×10^{-2}
frequency	3×10^{-2}
pressure and temperature correction	included in the Zener uncertainty budget
total	0.87

Table 3. Estimated standard uncertainties for a Zener calibration with the NIS equipment at the level of 10 V. The standard deviation of the mean of the NIS measurement results are 13 nV and 23 nV for BIPM_B and BIPM_C respectively. The contribution of the Zener noise is separately included in Table 1

Source of Uncertainty	**Type**	**Standard Uncertainty**
Voltage due to gain error of nanovoltmeter	**B**	**3 nV**
Voltage due to Thermal emf of polarity switch	**B**	**7 nV**
Voltage due to Leakage Current	**B**	**0.3 nV**
Uncertainty of Supravolt Control System	**A&B**	**4 nV**
Measured mean voltage (type A) ***Noise of the Fluke732B (1/f noise)***	***A***	***100 nV***
Fluke732B dependence on temperature, pressure, humidity	***B***	***100 nV***

Results at 1.018 V

Figure 3 shows the measured values obtained for the two standards by the two laboratories at 1.018 V and figure 4 presents the voltage evolution of the simple mean of the two standards which is used to compute the final result at 1.018 V. A linear least squares fit is applied to the results of the BIPM to obtain the results for both standards and their uncertainties at a common reference date corresponding to the mean date of the NIS measurements (2014/09/07).

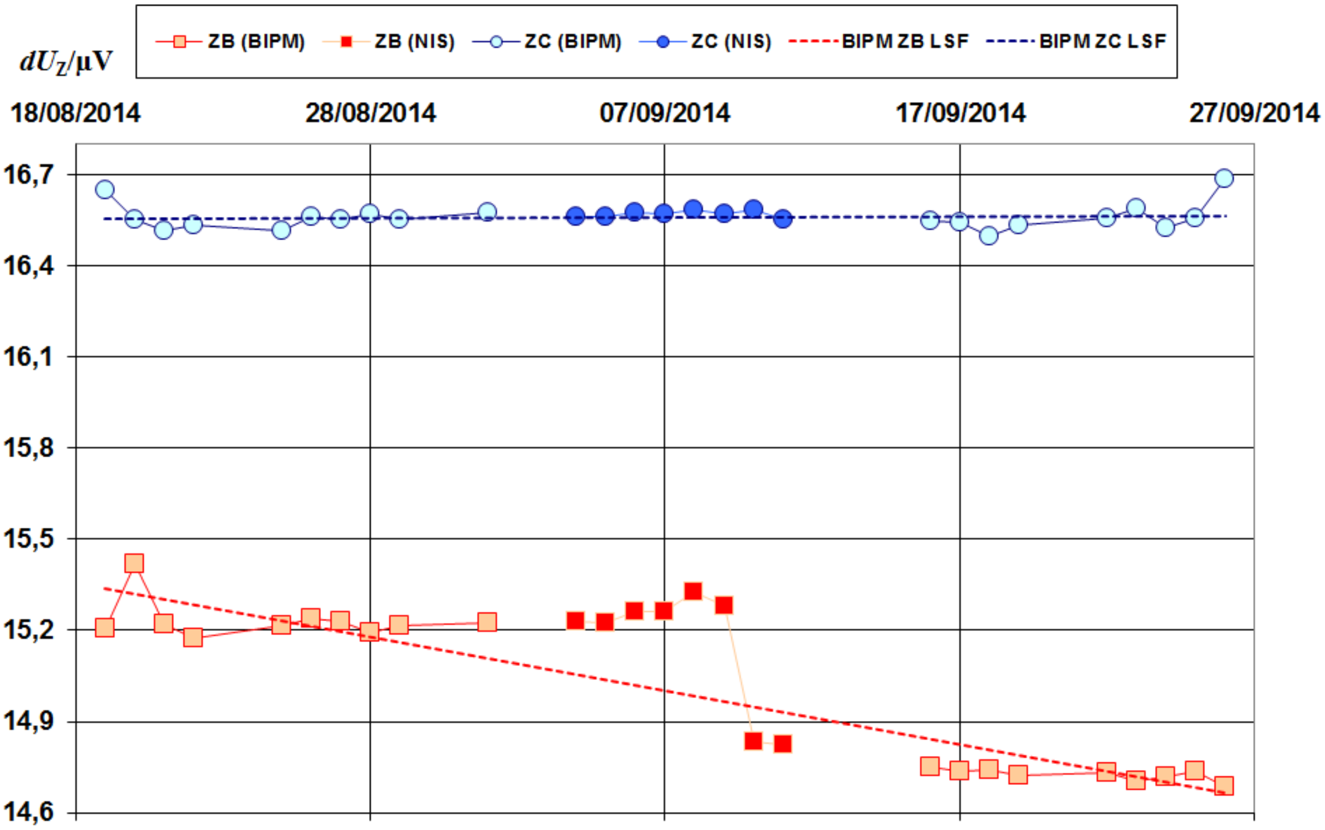

Figure 3: Voltage of BIPM_C (disks) and BIPM_B (squares) at 1.018 V measured at both institutes, referred to an arbitrary origin, as a function of time, with a linear least-squares fit to the measurements of the BIPM.

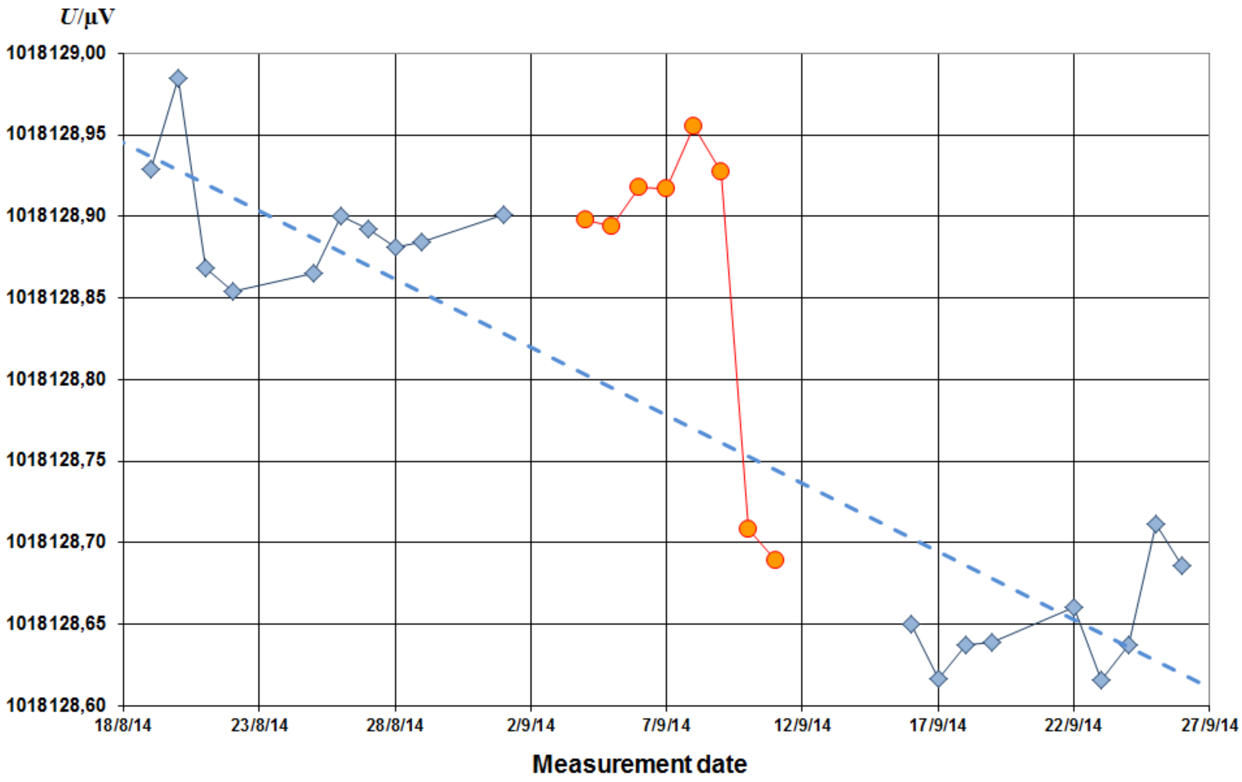

Figure 4: Voltage evolution of the simple mean of the two standards at 1.018 V. NIS measurements are represented by disks and BIPM measurements by filled diamonds.

Table 4 lists the results of the comparison and the uncertainty contributions for the comparison NIS/BIPM at 1.018 V. Experience has shown that flicker or 1/*f* noise ultimately limits the stability characteristics of Zener diode standards and it is not appropriate to use the standard deviation divided by the square root of the number of observations to characterize the dispersion of measured values. For the present standards, the relative value of the voltage noise floor due to flicker noise is about 1 part in 10^8.

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 and the "*a posteriori*" uncertainty as described for the measurements at 10 V.

Table 5 summarizes the uncertainties related to the calibration of a Zener diode against the Josephson array voltage standard at the BIPM and Table 6 lists the uncertainties related to the calibration of a Zener diode against the Josephson array voltage standard at the NIS.

(U_Z – 1.018 V)

Table 4. Results of the NIS (Egypt)/BIPM bilateral comparison of 1.018 V standards using two Zener traveling standards: reference date 07 September 2014. Uncertainties are 1 σ estimates.

		BIPM_B	BIPM_C
1	*NIS (Egypt)* (U_Z – 1.018 V)/µV	125.56	132.17
2	Type A uncertainty/µV	0.07	0.01
3	correlated unc. /µV	0.016	
4	*BIPM* (U_Z – 1.018 V)/µV	125.39	132.16
5	Type A uncertainty/µV	0.017	0.011
6	correlated unc./µV	0.001	
7	pressure and temperature correction uncertainty/µV	0.001	0.001
8	($U_{\mathrm{NIS}} - U_{\mathrm{BIPM}}$)/µV	0.16	0.01
9	uncorrelated uncertainty/µV	0.06	0.02
10	< $U_{\mathrm{NIS}} - U_{\mathrm{BIPM}}$ >/µV	0.088	
11	*a priori* uncertainty/µV	0.03	
12	*a posteriori* uncertainty/µV	0.08	
13	correlated uncertainty/µV	0.016	
14	comparison total uncertainty/µV	0.08	

In Table 4, the following elements are listed:

(1) the value attributed by NIS to each Zener U_{NIS}, computed as the simple mean of all data from NIS;

(2) the Type A uncertainty which is the larger of the experimental standard deviation of the mean of the measurements performed at NIS, and the 1/*f* noise floor of 10 nV which, according to the experience of the BIPM, in general limits the accuracy of Zener voltage standards [3].

(3) the uncertainty component arising from the maintenance of the volt at NIS: 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 the NIS measurements;

(7) the uncertainty due to the combined effects of the uncertainties of the pressure and temperature coefficients[*] and to the differences of the mean pressures and temperatures in the participating laboratories is calculated using the following assumption:

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[*] The evaluation of the correction coefficients was performed in 2000.

The uncertainty on the temperature correction $u_{T,i}$ of Zener i is determined for the difference ΔR_i between the mean values of thermistor resistances measured at both institutes which is then multiplied by the uncertainty $u(c_{T,i})$ of the temperature coefficients of this Zener standard:

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

where U = 1.018 V, $u(c_{\mathrm{T,ZB}}) = 0.522\times10^{-7}$ / kΩ, $u(c_{\mathrm{T,ZC}}) = 0.625\times10^{-7}$ / kΩ and ΔR_{ZB} = 0.008 kΩ and ΔR_{ZC} = 0.019 kΩ.

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

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

where U = 1.018 V, $\mathrm{u}(c_{\mathrm{P,ZB}}) = 0.063\times10^{-9}$ / hPa, $\mathrm{u}(c_{\mathrm{P,ZC}}) = 0.085\times10^{-9}$ / hPa, ΔP_{ZB} = 1.1 hPa and ΔP_{ZC} = 1.0 hPa.

The uncertainties on the measurement of the temperature and the pressure are negligible.

(8) the difference ($U_{\mathrm{NIS}} - U_{\mathrm{BIPM}}$) for each Zener, and (9) the uncorrelated part of the uncertainty, calculated as the quadratic sum of lines 2, 5 and 7;

(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,

(12) the *a posteriori* uncertainty;

(13) the correlated part of the uncertainty, calculated as the quadratic sum of lines 3 and 6, and

(14) the total uncertainty of the comparison, which is the root sum square of the correlated part of the uncertainty and of the larger of (11) and (12).

As the *a priori* uncertainty and the *a posteriori* uncertainty are different, the larger component is considered as the **transfer** uncertainty and is therefore equal to 80 nV.

The result of the comparison is presented as the difference between the value assigned to a 1.018 V standard by NIS, at NIS, U_{NIS}, and that assigned by the BIPM, at the BIPM, U_{BIPM}, which for the reference date is

$$\boldsymbol{U_{\mathrm{NIS}} - U_{\mathrm{BIPM}} = 0.09\ \mu\mathrm{V}; \qquad u_{\mathrm{c}} = 0.08\ \mu\mathrm{V} \quad \text{on 2014/09/07}},$$

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, (based on $K_{\mathrm{J-90}}$) and at NIS and the uncertainty related to the comparison.

Table 5. Estimated standard uncertainties for Zener calibrations with the BIPM equipment at the level of 1.018 V without the contribution of the Zener noise.

JVS & detector uncertainty components	Uncertainty/nV
Residual thermal electromotive forces	included in the Type A uncertainty
Noise of the measurement loop that includes the residual thermal electromotive forces including the residual EMF of the reversing switch	0.34
detector gain	0.11
leakage resistance	3×10^{-3}
frequency	3×10^{-3}
pressure and temperature correction	included in the Zener unc. budget
total	0.36

Table 6. Estimated standard uncertainties for Zener calibrations with the NIS equipment at the level of 1.018 V. The standard deviation of the mean of the NIS measurement results is in the interval from 72 nV to 4 nV for BIPM_B and BIPM_C respectively. The contribution of the Zener noise is separately included in Table 4

Source of Uncertainty	**Type**	**Standard Uncertainty**
Voltage due to gain error of nanovoltmeter	**B**	**3 nV**
Voltage due to Thermal emf of polarity switch	**B**	**7 nV**
Voltage due to Leakage Current	**B**	**10 nV**
Uncertainty of Supravolt Control System	**A&B**	**4 nV**
Measured mean voltage (type A) ***Noise of the Fluke732B (1/f noise)***	***A***	***not lower than the 1/f noise floor (10 nV)***
Fluke732B dependence on temperature, pressure, humidity	***B***	***10 nV***

Conclusion

The final result of the comparison is presented as the difference between the values assigned to DC voltage standards by NIS, at the level of 1.018 V and 10 V, at NIS, U_{NIS}, and those assigned by the BIPM, at the BIPM, U_{BIPM}, at the reference date of the 7th of September 2014.

$\boldsymbol{U_{\mathrm{NIS}} - U_{\mathrm{BIPM}} = 0.09\ \mu\mathrm{V}}$; $\boldsymbol{u_{\mathrm{c}} = 0.08\ \mu\mathrm{V}}$, **at 1 V**

$\boldsymbol{U_{\mathrm{NIS}} - U_{\mathrm{BIPM}} = 0.22\ \mu\mathrm{V}}$; $\boldsymbol{u_{\mathrm{c}} = 0.14\ \mu\mathrm{V}}$, **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 NIS, based on $K_{\mathrm{J-90}}$, and the uncertainty related to the comparison.

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

References

[1] Josephson Voltage Standard SupraVOLTcontrol Manual, Supracon AG, Wildenbruchstr 15, 07745 Jena, Germany, 2013.

[2] M. Starkloff, G. Wende, S. Anders, M. Schubert, M. Meyer and H.-G. Meyer: Operation and Accuracy of an Automated Cryocooler-based 10 Volt Josephson Voltage Standard System, IEEE/CSC & ESAS EUROPEAN SUPERCONDUCTIVITY NEWS FORUM (ESNF), No. 16, April 2011.

[3] Witt, T.J., Maintenance and dissemination of voltage standards by Zener-diode-based instruments, IEE Proc. Sci. Meas. Technol., 149(6), pp 305-312, November 2002.