# Bilateral Comparison of 1 V and 10 V Standards between the DMDM (Serbia) and the BIPM, January to March 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 Directorate of Measures and Precious Metals (DMDM), Beograd, Serbia, was carried out from January to March 2014. Two BIPM Zener diode-based travelling standards (Fluke 732B), BIPM\_6 (Z6) and BIPM\_A (ZA), were transported by freight to DMDM. At DMDM, 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 DMDM, 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

## DMDM 1 V and 10 V measurements

DMDM maintains a DC voltage primary standard based on the Josephson Effect: SupraVOLTcontrol system, LHe-free version cooled with a cryocooler, serial no.12, Supracon AG [1,2].

The system includes the following:

- a) 10 volt SIS Josephson series array mounted in cryocooler with serial no.12, manufacturer Institute of Photonic Technology, serial number 1996-1
- b) Nanovoltmeter, 2182A, Keithley
- c) EIP microwave source locking counter
- d) Supracon control electronic unit
- e) Switchable power socket
- f) TransMIT pulse tube cooler including 10 V Josephson voltage standard circuit
- g) Filter box
- h) Vacuum relief pressure valve
- i) Rotary valve with shielding and housing
- j) Vacuum pump CDK180 including pressure sensor, vacuum valve, KF25 tube
- k) Toshiba frequency converter including cables
- l) Compressor
- m) Aeroquip flexlines box, with 2 pieces each 9 m long, including filter cartridge
- n) GPS 10 MHz reference frequency unit box including receiver, antenna
- o) 75 GHz microwave electronics box including Gunn-oscillator, isolator, directional coupler, remote sensor, voltage controlled attenuator
- p) Polarity reversal switch box including cables
- q) Host computer including laptop, accumulator, windows 7, driver CD and supraVOLTcontrol system software

#### SPECIFICATIONS OF THE SYSTEM:

Typical calibration accuracy (direct comparison to a second Josephson voltage standard)  $\pm 4 \text{ nV} @ 10 \text{ V}, \Delta V/V(10\text{ V}) = 4 \times 10^{-10}$ 

Typical calibration accuracy of secondary voltage standards (type A) for all three channels (limited by the noise of the secondary voltage standard):

 $\pm 20 \text{ nV} = 1 \text{ V}, \ \Delta V / V (1 \text{ V}) = 2 \times 10^{-8}$ 

 $\pm 100 \text{ nV}$  @ 10 V,  $\Delta V/V(10\text{V}) = 1 \times 10^{-8}$ 

Typical calibration accuracy of the gain factor of external voltmeters

(depends on the type of voltmeter)

 $\Delta g/g < 2 \times 10^{-6}$ 

Step flatness = 0 m $\Omega$ test measurements are limited by the noise of the secondary voltage standard:

Standard.
< 25 m  $\Omega$  @ 10 V
< 10 m  $\Omega$  @ 1 V
Isolation resistance for all channels
> 100 G  $\Omega$  @ Low – High
> 50 G  $\Omega$  @ Low – Ground
> 50 G  $\Omega$  @ High – Ground
Leakage current at 10 V for all channels
< 100 p A @ Low – High</p>

< 200 pA @ Low – Ground</p>
< 200 pA @ High – Ground</p>
Thermal voltage of the reversal switch including the connecting cables
< 25 nV, typically below 5 nV for each channel</p>
Accuracy of the 10 MHz reference frequency  $\Delta f/f < 1 \times 10^{-10}$  (option external 10 MHz reference frequency)
Stability of the 75 GHz Gunn oscillator (locked by the EIP source locking microwave counter):
± 10 Hz @ 75 GHz  $\Delta f/f < 1.4 \times 10^{-10}$ Gain error of the null detector (see also the specification of the Keithley 2182 voltmeter)
1.00003 @ 10 mV range
Accuracy of the Sensors:
± 0.5 K for a temperature of 0°C to 30°C
± 2 % for a relative humidity of 10 % to 100 %
± 1 mbar for an air pressure of 800 mbar to 1100 mbar

The system automatically calibrates secondary voltage standards. Altogether 8 data points were measured with their standard deviation and offset voltages in the wiring loop. Each data point consists of 20 readings of the voltage difference between the Fluke 732B voltage and the Josephson voltage read out by the integrated nanovoltmeter, in positive and negative polarity. The driving frequency is displayed as the mean value of 20 readings together with their simple standard deviation and the data of the integrated sensors for environment temperature, barometric pressure and humidity. The calculated output voltages of the Fluke 732B are displayed with their standard deviations [1,2] (Cf. following paragraphs).

The thermistor resistance measurements were carried out with a HP3458A digital multimeter, (S/N 2823A18556). The setup of the ohmmeter was as follows: Range: 1 M $\Omega$  (OHMF, OCOMP ON, DELAY 1), Current source: 5  $\mu$ A.

## BIPM Measurements for both 1 V and 10 V

The output voltage of the Zener standard to be measured is connected to the BIPM Josephson Voltage Standard - Hypres 10 V SIS array (S/N: 2538E-7) - 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 at least two hours after 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.

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  $\mu$ V for both nominal voltages. The nanovoltmeter is set to its 3  $\mu$ V range for the measurements performed at the level of 1 V and on its 10  $\mu$ V 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 microvolts).

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 to many bips

occur, the operator can start the "Data Acquisition" sequence over. 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).

### Additional measurements at 10 V

BIPM received a 10 V SNS based programmable array from PTB [3] very recently and decided to implement this array into the automatic measurement setup we operate at the 1 V level since 2007 [4]. The performances of this measurement setup at the 10 V level could be tested during the comparison return measurements, fully independently from the official SIS-BIPM measurement unit. The results are presented in the next paragraph and more details are given in the Appendix A1.

## 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 DMDM measurements (2014/02/13).



Figure 1a: Voltage of Z6 (top) and ZA (bottom) at 10 V measured at both institutes (squares for BIPM and disks for DMDM) referred to an arbitrary origin as a function of time, with a linear least-squares fit adjustment to the BIPM measurements. The green crosses are independent measurements of the two standards carried out at BIPM on the return of the standards carried out with an SNS JVS (see Appendix A).





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

Table 1. Results of the DMDM (Serbia)/BIPM bilateral comparison of 10 V standards using two Zener
traveling standards: reference date 13 February 2014. Uncertainties are 1 $\sigma$ estimates.

		BIPM_6	BIPM_A
1	<i>DMDM (Serbia)</i> ( <i>U</i> <sub>z</sub> – 10 V)/μV	-55.08	-27.42
2	Type A uncertainty/µV	0.167	0.100
3	correlated (Type B) unc. /µV	0.	018
4	<i>BIPM</i> ( <i>U</i> <sub>Z</sub> – 10 V)/μV	-55.36	-27.92
5	Type A uncertainty/µV	0.1	0.1
6	correlated (Type B) unc./µV	0.	001
7	pressure and temperature correction uncertainty/µV	0.14	0.02
8	$(U_{\text{DMDM}} - U_{\text{BIPM}})/\mu V$	0.277 0.494	
9	uncorrelated uncertainty/µV	0.24	0.14
10	$< U_{\rm DMDM} - U_{\rm BIPM} > /\mu V$	0.	385
11	<i>a priori</i> uncertainty/µV	0.	111
12	<i>a posteriori</i> uncertainty/µV	0.	108
13	correlated uncertainty/µV	0.	018
14	comparison total uncertainty/µV	0.	112

In Table 1, the following elements are listed:

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

(2) the Type A uncertainty which is the experimental standard deviation of the measurements performed at DMDM;

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

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

The uncertainty on 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 uncertainty  $u(c_{T,i})$  of the temperature coefficient of this Zener standard:

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

where U = 10 V,  $u(c_{T,Z6}) = 1.03 \times 10^{-7}$  / k $\Omega$ ,  $u(c_{T,ZA}) = 0.39 \times 10^{-7}$  / k $\Omega$  and  $\Delta R_{Z6} = 0.138$  k $\Omega$  and  $\Delta R_{ZA} = 0.060$  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:

$$U_{\rm P,i} = U \times U(C_{\rm P-i}) \times \Delta P_i$$

where U = 10 V,  $u(c_{P,Z6}) = 0.08 \times 10^{-9}$  / hPa,  $u(c_{P-ZA}) = 0.082 \times 10^{-9}$  / hPa,  $\Delta P_{Z6} = 2.9$  hPa and  $\Delta P_{ZA} = 0.3$  hPa.

The uncertainty on the measurement of the temperature is negligible. After the comparison results were communicated to the participant, DMDM informed BIPM that the embedded atmospheric pressure gauge of their JVS was found defective. A proposal for a corrected result is presented in the Appendix B of the report.

(8) the difference  $(U_{\text{DMDM}} - U_{\text{BIPM}})$  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 described in the following note.

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

<u>Note</u>: The *a posteriori* uncertainty is the standard deviation of the mean of the voltage difference of each individual transfer standard and the *a priori* uncertainty is the quadratic combination of the Type A uncertainties of the two labs.

(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

<sup>&</sup>lt;sup>\*</sup> The evaluation of the correction coefficients was performed in 1997.

standard deviation of the mean of the results from the two traveling standards<sup>\*</sup>. If the "*a posteriori*" uncertainty is significantly larger than the "*a priori*" uncertainty, we assume that a 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 DMDM, at DMDM,  $U_{DMDM}$ , and that assigned by the BIPM, at the BIPM,  $U_{BIPM}$ , which for the reference date is

## $U_{\text{DMDM}} - U_{\text{BIPM}} = 0.39 \ \mu\text{V}; \quad u_{\text{c}} = 0.11 \ \mu\text{V} \quad \text{ on } 2014/02/13,$

where  $u_c$  is the combined standard uncertainty associated with the measured difference, including the uncertainty of the representation of the volt at DMDM, at the BIPM (based on  $K_{J-90}$ ), 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.

Tables 3a and 3b list the uncertainties related to the calibration of a Zener at the DMDM. 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).

<sup>&</sup>lt;sup>\*</sup> 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.

# **Uncertainty Budgets**

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

JVS & detector uncertainty components	Uncertainty/nV
Noise of the measurement loop that includes the	0.86
residual thermal electromotive forces including	
the residual EMF of the reversing switch	
detector gain	0.11
leakage resistance	3×10 <sup>-2</sup>
frequency	3×10 <sup>-2</sup>
pressure and temperature correction	included in the Zener unc. budget
total	0.87

# Table 3. Estimated standard uncertainties for Zener calibrations with the DMDM equipment at the level of 10 V for each Zener.

Table 3a (ZA).

Quantity	Estimate	Туре	Uncertainty (nV)	Note
Uncertainty of supraVOLTcontrol system	10 V	A & B	2	
Measured mean voltage (type A), noise of the zener (1/f noise)	10 V	А	100	А
Voltage due to gain error of the nanovoltmeter	250 μV	В	10	В
Voltage due to leakage current ( $R_{ISO}=10$ G $\Omega$ of the zener)	1.7 nV (corresponding to a leakage current of <i>I</i> =1 nA)	В	1,7	С
Voltage due to thermal EMF of the polarity switch	0 V	В	5	D
Zener dependence on temperature, pressure, humidity	(TC < 400 nV/K)	В	15	Е
Electromagnetic interference	none		0	F
Total (RMS) extended combined uncertainty (k=2 -> 95% confidence)				

#### \*R = 3 $\Omega$ + 0,001 $\Omega \approx 3 \Omega$

Estimated \*R = 3  $\Omega$  is the output resistance of the Fluke standard (5m $\Omega$ ), additionally the total resistance of the wires (3 Ohm) in the measuring loop.

<u>Note</u>: There is somewhere in the circuit a limited isolation resistance to ground (e.g. 10 GOhms (typically) at the Fluke 732B or nanovoltmeter or cryoprobe), which causes a voltage drop at the measurement leads resistance (the maximum resistance in the measurement loop due to the wires in cryoprobe, polarity reversal, switch and Fluke 732B is about 3  $\Omega$ ). As the path of the leakage current is unknown, we assumed a rectangular distribution (uncertainty is divided by square root of 3)

## Table 3b (Z6)

Quantity	Estimate	Туре	Uncertainty (nV)	Note
Uncertainty of supraVOLTcontrol system	10 V	A & B	2	
Measured mean voltage (type A), noise of the zener (1/f noise)	10 V	А	167	А
Voltage due to gain error of the nanovoltmeter	250 μV	В	10	В
Voltage due to leakage current ( $R_{ISO}=10$ G $\Omega$ of the zener)	1.7 nV (corresponding to a leakage current of <i>I</i> =1 nA)	В	1,7	С
Voltage due to thermal EMF of the polarity switch	0 V	В	5	D
Zener dependence on temperature, pressure, humidity	(TC < 400 nV/K)	В	13	Е
Electromagnetic interference	none		0	F
Total (RMS) extended combined uncertainty ( $k=2 \rightarrow 95\%$ confidence)			335	

Note	
А	Typical range of uncertainty for different Fluke 732B, calculated for 8 data points.
В	Maximal voltage applied to the nanovoltmeter, the gain factor is typically in the range of $g = 1.00004$
С	Estimation of the voltage error due to leakage current at the wiring and the internal resistance of the Fluke standard ( $\Delta U = 10V \times R/R$ iso, with *R=3 $\Omega$ , Riso=10 G $\Omega$ @ 10V)
D	Typical thermal EMFs of the polarity switch
E	Must be measured and estimated for each Zener device individually
F	Measurements are performed in Faraday cage

## 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 DMDM measurements (2014/02/13).



Figure 3: Voltage of BIPM\_A (on top) and BIPM\_6 (on bottom) 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.

Table 4 lists the results of the comparison and the uncertainty contributions for the comparison DMDM/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 which consists of the experimental standard deviation of the mean of the results from the two traveling standards. Then we applied the same methodology as described in 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 DMDM. (*U*<sub>z</sub> – 1.018 V)

Table 4. Results of the DMDM (Serbia)/BIPM bilateral comparison of 1.018 V standards using two Zener traveling standards: reference date 13 February 2014. Uncertainties are 1  $\sigma$  estimates.

		BIPM_6	BIPM_A
1	<i>DMDM (Serbia)</i> ( <i>U</i> <sub>Z</sub> – 1.018 V)/μV	139.12	188.40
2	Type A uncertainty/µV	0.036	0.015
3	correlated unc. /µV	0.	059
4	<i>BIPM</i> ( <i>U</i> <sub>Z</sub> – 1.018 V)/μV	138.98	188.34
5	Type A uncertainty/µV	0.010	0.011
6	correlated unc./µV	0.	001
7	pressure and temperature correction uncertainty/µV	0.001	0.004
8	$(U_{\text{DMDM}} - U_{\text{BIPM}})/\mu V$	0.14 0.05	
9	uncorrelated uncertainty/µV	0.038	0.019
10	$< U_{\rm DMDM} - U_{\rm BIPM} > /\mu V$	0.	094
11	<i>a priori</i> uncertainty/µV	0.	021
12	a posteriori uncertainty/µV	0.	042
13	correlated uncertainty/µV	0.	059
14	comparison total uncertainty/µV	0.	072

In Table 4, the following elements are listed:

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

(2) the Type A uncertainty due to the instability of the Zener at DMDM;

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

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

The uncertainty on the temperature correction  $u_{T,i}$  of Zener *i* is determined for the difference  $\Delta R_i$ 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:

<sup>&</sup>lt;sup>\*</sup> The evaluation of the correction coefficients was performed in 1997.

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

where U = 1.018 V,  $u(c_{T,Z6}) = 0.08 \times 10^{-7} / k\Omega$ ,  $u(c_{T,ZA}) = 0.70 \times 10^{-7} / k\Omega$  and  $\Delta R_{Z6} = 0.117$  k $\Omega$  and  $\Delta R_{ZA} = 0.057$  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:

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

where U = 1.018 V,  $u(c_{P,Z6}) = 0.035 \times 10^{-9}$  / hPa,  $u(c_{P,ZA}) = 0.043 \times 10^{-9}$  / hPa,  $\Delta P_{Z6} = 2.8$  hPa and  $\Delta P_{ZA} = 2.7$  hPa.

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

(8) the difference  $(U_{DMDM} - U_{BIPM})$  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 42 nV. However, comparing the results obtained at BIPM before the shipment of the Zeners and after their return, it seems not obvious to conclude that the metrological quality of the standards was affected by their shipment.

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

#### $U_{\text{DMDM}} - U_{\text{BIPM}} = 0.094 \ \mu\text{V}; \ u_{\text{c}} = 0.072 \ \mu\text{V}$ on 2014/02/13,

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  $K_{J-90}$ ) and at DMDM 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	0.34
the residual thermal electromotive forces	
including the residual EMF of the reversing	
switch	
detector gain	0.11
leakage resistance	3×10 <sup>-3</sup>
frequency	3×10 <sup>-3</sup>
pressure and temperature correction	included in the Zener unc. budget
total	0.36

Table 6a and 6b. Estimated standard uncertainties for Zener calibrations with the DMDM for BIPM\_6 and BIPM\_A respectively at the level of 1.018 V. The standard deviation of the mean of the DMDM daily measurement results is equal to 19 nV.

Quantity	Estimate	Туре	Uncertainty (nV)	Note
Uncertainty of supraVOLTcontrol system	1.018 V	A & B	0,5	
Measured mean voltage (type A), noise of the zener (1/f noise)	1.018 V	А	32	А
Voltage due to gain error of the nanovoltmeter	250 μV	В	10	В
Voltage due to leakage current ( $R_{ISO}=10$ G $\Omega$ of the zener)	0.1 nA	В	58	С
Voltage due to thermal EMF of the polarity switch	0 V	В	5	D
Zener dependence on temperature, pressure, humidity	(TC < 400 nV/K)	В	2	Е
Electromagnetic interference	none		0	F
Total (RMS) extended combined uncertainty (k=2 -> 95% confidence)			135	

#### Table6a (Z6)

#### Table 6b (ZA)

Quantity	Estimate	Туре	Uncertainty (nV)	Note
Uncertainty of supraVOLTcontrol system	1.018 V	A & B	0,5	
Measured mean voltage (type A), noise of the zener (1/f noise)	1.018 V	А	14	А
Voltage due to gain error of the nanovoltmeter	250 μV	В	10	В
Voltage due to leakage current ( $R_{ISO}=10$ G $\Omega$ of the zener)	0.1 nA	В	58	С
Voltage due to thermal EMF of the polarity switch	0 V	В	5	D
Zener dependence on temperature, pressure, humidity	(TC < 400 nV/K)	В	3	Е
Electromagnetic interference	none		0	F
Total (RMS) extended combined uncertainty (k=2 -> 95% confidence)			122	

А	Typical range of uncertainty for different Fluke 732B, calculated for 8 data points.
В	Maximal voltage applied to the nanovoltmeter, the gain factor is typically in the range of $g = 1.00004$
С	Estimation of the voltage error due to leakage current and the internal resistance of the Fluke standard ( $\Delta U = 1V \times \text{Rinternal/Riso}$ , with Rinternal=1 k $\Omega$ , (at 1 V the internal resistance of the Fluke 732B is 1k $\Omega$ !) Riso=10 G $\Omega$ ( <i>a</i> 1V)
D	Typical thermal EMFs of the polarity switch
Е	Must be measured and estimated for each Zener device individually
F	Measurements are performed in Faraday cage

## Conclusion

The final result of the comparison is presented as the difference between the values assigned to DC voltage standards by DMDM, at the level of 1.018 V and 10 V, at DMDM,  $U_{\text{DMDM}}$ , and those assigned by the BIPM, at the BIPM,  $U_{\text{BIPM}}$ , at the reference date of the 13<sup>th</sup> of February 2014.

 $U_{\text{DMDM}} - U_{\text{BIPM}} = 0.094 \ \mu\text{V}; \ u_{\text{c}} = 0.072 \ \mu\text{V}, \text{ at 1 V}$ 

 $U_{\text{DMDM}} - U_{\text{BIPM}} = 0.39 \ \mu\text{V}; \quad u_{\text{c}} = 0.12 \ \mu\text{V}, \text{ at 10 V}$ 

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 DMDM, based on  $K_{J-90}$ , and the uncertainty related to the comparison.

The results at the 10 V level are not covered by the uncertainties with a coverage factor of 2. After the distribution of the Draft A, the DMDM discovered that the pressure gauge was defective. Some considerations on the correction to apply on the comparison result and the corresponding uncertainties are presented in the Appendix B.

Nevertheless, the above results fully cover the CMCs of DMDM which are significantly larger. No corrections for temperature and pressure are applied in calibrations for customers' secondary standards.

BIPM noticed that DMDM measurements all appear slightly above the fitting line of the BIPM measurements at 1 V and 10 V, for both transfer standards. In such a case, one would suspect a systematic error in the measurement loop.

Moreover, the DMDM commercial JVS system operates with the measurement loop floating from the ground potential. This might be adapted to specific environmental conditions but might also expose the setup to pick-up electromagnetic noise in other experimental conditions.

It might be interesting to perform in the future some Zener measurements with the low potential side of the array grounded and to compare the results with the configuration where the array is floating from the ground.

## 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 planned to be upgraded for operation up to 10 V. The 1 V system was based on a PTB programmable array made of SINIS (Superconductor/Isolator/Normal Metal/Isolator/ Superconductor) Josephson junctions and will be replaced with a 10 V PTB programmable array based on the new SNS (Superconductor/Normal Metal/Superconductor) junctions technology [5].

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

	Z6 / µV	ZA / µV
mean value (7 meas. days) - SIS	9999944,265	9999971,968
std. dev. of the mean - SIS	0,072	0,094
mean value (7 meas. days) - SNS	9999944,294	9999972,108
std. dev. of the mean - SNS	0,058	0,088
"SNS" – "SIS"	0,029	0,140

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

If the BIPM return measurements are carried out with the SNS-based measurement setup, the computation on the comparison result give a result of  $U_{DMDM} - U_{BIPM} = 0.36 \,\mu\text{V}$  – as compared to 0.39  $\mu$ V with the SIS-array - with the same total combined uncertainty at 10 V. This result shows that the two fully independent BIPM measurements setups are equivalent within the stated uncertainties.

# Appendix B: Failure of the embedded pressure gauge of the DMDM Josephson Voltage Standard.

After DMDM had been informed of the comparison results, the personnel involved in the comparison was concerned that the uncertainties were not covering the 10 V result with a coverage factor of k=2.

DMDM has then been in contact with their JVS manufacturer to investigate on possible measurement errors arising from the primary voltage standard. The following is the chronological list of the steps of the investigation process:

### 1) Excess of noise:

a) The manufacturer recommended to look for excess noise on the basis that the standard deviation of the measured voltage would typically be below 500 nV at 10 V (at 1 V below 200 nV), for a 732A/B Zener voltage standard.

#### DMDM noted that all the comparison results were measured below this noise floor.

b) The assumption of a possible systematic error introduced by a ground loop was discarded, as the Josephson array is completely floating from ground during the time of data acquisition. The nanovoltmeter HI - and LO inputs are floating, as well as the 732B outputs. (The floating Josephson voltage is a speciality of the Supracon system, realised by the use of zero current Shapiro steps and the disconnection of the Josephson array from its biasing source by relays).

# DMDM applied the measurement procedure according to the operating manual from Supracon.

c) The manufacturer recommended to test different grounding connections (Zener connected to the mains or on batteries) including some checks on the quantum behaviour of the array within the tested configuration.

DMDM checked the array conditions before each measurements and found the array within the accepted range. DMDM also performed measurements with their own FLUKE 732B (April 2014) and checked that the connections could not be the reason of a leakage resistance to ground error. No significant difference was recorded in those tests. The measurements were all within the expected level of the noise. DMDM staff visited the manufacturer where a Zener Fluke 732A was calibrated using their Josephson system, including different types of connections of the Zener to the scanner. No discrepancies a the noise floor level were revealed. No systematic error could be identified.

## 2) Frequency:

- a) The correct operation of the microwave counter was checked with a 1 MHz external reference signal.
- **b)** The 10 MHz reference frequency which is stabilized and traced back to the GPS satellites was also checked.

### 3) Polarity reversal switch:

The polarity reversal switch that changes the polarity of the Zener standard, in order to cancel out offset voltages and thermal EMFs in the loop, was controlled. The residual voltage at the level of the polarity reversal switch is to be below 5 nV.

DMDM checked offset voltages and thermal EMFs in the loop according to the advice from manufacturer.

#### 4) Nanovoltmeter:

The nanovoltmeter measures the difference voltage between the Josephson voltage standards and the Zener on its 10 mV range. An error on the voltage reading can possibly come from the gain error. The gain is measurable by the "Performance Test" from a dedicated software "CALIBRATION NANOVOLTMETER" provided by the manufacturer.

During the comparison, DMDM performed a gain calibration of the nanovoltmeter, prior to the Zener calibration. The new detector gain was saved to the software configuration file.

#### 5) Leakage current:

In principal the effect of a leakage current can be measured by adding an additional resistor in the wiring loop, as due to the voltage divider the measured voltage of the 732B would decrease in this case. From the comparison results, the Zener voltage measured by DMDM was higher than that measured by BIPM, therefore this possible source of error was rejected.

## 6) 732B dependencies on temperature, humidity, pressure, drift (e.g. transportation):

The temperature and pressure sensors embedded in the JVS were controlled by comparison with measurements of the ambient laboratory temperature and pressure by a calibrated independent thermometer and barometer.

The embedded barometer readings were found to be wrong by 1.5 hPa. In addition, some of the pressure measurements performed during the comparison were found discrepant: the pressure recorded (for the Fluke s/n 7480002) on the 14<sup>th</sup> of February at 12:13 was 984 hPa and at 12:21 (8 minutes later) was 1000 hPa.

The manufacturer also suggested to take into consideration the possibility of excluding some measurements that were collected, because of an excessive level of noise. DMDM decided to not remove any measurement as the noise range was always below the accepted limit of 500 nV.

The comparison was completed within a two months period and it is reasonable to discard any long term seasonal effect due to the relative humidity level in air.

The comparison result at 10 V was re-calculated after the application of a correction of 1.5 hPa to the DMDM pressure measurements together with an increase of the uncertainty on the pressure measurement by a factor of 10 as the sensor seems to be not fully reliable. The corresponding result of the comparison at 10 V would then be:

## $U_{\text{DMDM}} - U_{\text{BIPM}} = 0.33 \ \mu\text{V}; \quad u_{\text{c}} = 0.12 \ \mu\text{V}, \text{ at 10 V}$

The corresponding updated uncertainty budget is presented in Table B1 based on the new pressure uncertainty and the equation  $u_{P,i} = U \times u(c_{P,i}) \times \Delta P_i$ 

		BIPM_6	BIPM_/	4
1	DMDM (Serbia)	-55.12	-27.5	0
	( <i>U</i> <sub>Z</sub> – 10 V)/μV			
2	Type A uncertainty/µV	0.167	0.100	
3	correlated (Type B) unc. /µV	0.018		
4	<i>BIPM</i> ( <i>U</i> <sub>Z</sub> – 10 V)/μV	-55.36	-27.92	
5	Type A uncertainty/µV	0.1	0.1	
6	correlated (Type B) unc./µV	0.001		
7	pressure and temperature	0.148	0.0	43
	correction uncertainty/µV			
8	$(U_{\text{DMDM}} - U_{\text{BIPM}})/\mu V$	0.244	0.411	
9	uncorrelated uncertainty/µV	0.244	0.148	
10	$< U_{\rm DMDM} - U_{\rm BIPM} > /\mu V$	0.	0.328	
11	a priori uncertainty/µV	0.120		
12	a posteriori uncertainty/µV	0.084		
13	correlated uncertainty/µV	0.018		
14	comparison total uncertainty/µV	0.12		

The same process is considered at the level of 1 V with an increase of the pressure uncertainty by a factor of 10 from which we can derive the following uncertainty budget (Table B2) at 1  $\sigma$  estimates

		BIPM_6	BIPM_	_A	
1	<i>DMDM (Serbia)</i> ( <i>U</i> <sub>Z</sub> – 1.018 V)/μV	139.12	188.39		
2	Type A uncertainty/µV	0.036	0.015		
3	correlated unc. /µV	0.059			
4	<i>BIPM</i> ( <i>U</i> <sub>Z</sub> – 1.018 V)/μV	138.98	188.34		
5	Type A uncertainty/µV	0.010	0.011		
6	correlated unc./µV	0.001			
7	pressure and temperature	0.002	0.0	004	
	correction uncertainty/µV				
8	$(U_{\text{DMDM}} - U_{\text{BIPM}})/\mu V$	0.143	0.05		
9	uncorrelated uncertainty/µV	0.038	0.019		
10	$< U_{\rm DMDM} - U_{\rm BIPM} > /\mu V$	0.	0.097		
11	a priori uncertainty/µV	0.021			
12	a posteriori uncertainty/µV	0.042			
13	correlated uncertainty/µV	0.			
14	comparison total uncertainty/µV	0.072			

The comparison result at 1 V was re-calculated after the application of a correction of 1.5 hPa to the DMDM pressure measurements together with an increase of the uncertainty on the pressure measurement is:

 $U_{\text{DMDM}} - U_{\text{BIPM}} = 0.097 \ \mu\text{V}; \ u_{\text{c}} = 0.072 \ \mu\text{V}, \text{ at 1 V}$ 

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