Rapport BIPM-2007/07

# Bilateral Comparison of 10 V Standards between the VNIIM (Russia) and the BIPM, August to October 2007 (part of the ongoing BIPM key comparison BIPM.EM-K11.b)

by A. S. Katkov\*\*, S. Solve\* and M. Stock\*

\*Bureau International des Poids et Mesures, F-92312 Sèvres Cedex

\*\* Mendeleyev Institute for Metrology (VNIIM), Moskovsky pr.19,

190005 St.Petersburg, Russia



## Bilateral Comparison of 10 V Standards between the VNIIM (Russia) and the BIPM, August to October 2007 (part of the ongoing BIPM key comparison BIPM.EM-K11.b)

by A. S. Katkov\*\*, S. Solve\* and M. Stock\*

\*Bureau International des Poids et Mesures, F-92312 Sèvres Cedex

\*\* Mendeleyev Institute for Metrology (VNIIM), Moskovsky pr.19,

190005 St. Petersburg, Russia

As a 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 Mendeleyev Institute for Metrology (VNIIM), St. Petersburg, Russia, was carried out from August to October 2007. A Zener-diode based travelling standard was calibrated by the Josephson voltage standards of both institutes. The comparison measurement sequence was VNIIM – BIPM – VNIIM. The travelling standard (Fluke 732B - S/N 7590806) was provided by VNIIM and was transported by hand.

<u>Measurements at VNIIM</u>: The VNIIM measurements were carried out by comparison with the VNIIM primary Josephson voltage standard [1], which comprises four different main parts:

1) A microwave source referenced to a 5 MHz signal produced by a rubidium frequency standard;

2) A cryoprobe (20 mm stainless steel tube) equipped with a waveguide to which a 10 V PTB Josephson SIS array (S/N: Me 168/9) is connected. The probe is immersed in a liquid helium dewar. The measurement leads are protected against EMI with LF filters;

3) A laboratory-made current bias source with a variable internal resistance (30 to 1000  $\Omega$ ) is used to bias the array and to select the voltage step. An oscilloscope is connected to the bias circuit to monitor the /-*V* characteristic of the array;

4) A digital nanovoltmeter (Keithley 2182) equipped with a polarity reversal switch monitors the voltage difference between the reference and the transfer standard. The data are recorded using computer software.

More details on the VNIIM measurement system together with the measurement method are given in Annex A.

<u>Measurements at BIPM</u>: At the BIPM, the travelling standard was calibrated with the Josephson voltage standard regularly used for the BIPM.EM-K10 and BIPM.EM-K11 comparisons [2].

**<u>Results</u>**: The atmospheric pressure and the internal thermistor resistance were measured and reported for each measurement of the output voltage, at the VNIIM and at the BIPM. The measurement results of the VNIIM were sent without applying any correction for temperature or atmospheric pressure. The BIPM applied the corrections for temperature and atmospheric pressure on all results using the following formula:

$$U_0 = U_{\rm m} \times (1 - C_{\rm T} \times (R_{\rm m} - R_0) - C_{\rm P} \times (P_{\rm m} - P_0))$$

where  $U_0$  is the corrected value,  $U_m$  the measured value,  $C_T$  and  $C_P$  the temperature and pressure coefficients,  $R_m$  and  $P_m$  the values of the thermistor resistance and atmospheric pressure at the time of the measurement,  $R_0$  the thermistor reference value and  $P_0$  the reference atmospheric pressure.

Standard	Output	Thermistor reference value <i>R</i> ₀ / kΩ	Temperature coefficient / kΩ	Pressure reference value <i>P</i> <sub>0</sub> / hPa	Pressure coefficient / hPa
ZA	10 V	39.2	1.9 × 10 <sup>-7</sup>	1013.25	1.17 × 10 <sup>-9</sup>

The ambient temperature and relative humidity were also measured and indicated in the results' form. No corrections were applied for the variations of these parameters. Figure 1 shows the measured values, after correction, obtained for the standard by the two laboratories. A linear least squares fit is applied to the results of the VNIIM to obtain the value for the standard and its uncertainty at a common reference date.



**Figure 1**. Voltage of the VNIIM Zener Standard (S/N 7590806) at 10 V measured at both institutes, as a function of time, with linear least-squares fit to the measurements of the VNIIM.

Table 1 lists the results of the comparison and the uncertainty contributions for the comparison VNIIM/BIPM. BIPM experience has shown that flicker or 1/f noise ultimately limits the stability characteristics of Zener diode standards and that it is not appropriate to use the standard deviation divided by the square root of the number of observations to characterize the standard deviation of the mean. For Zener standards, the relative value of the voltage noise floor due to flicker noise, attributed by BIPM, is at least 1 part in  $10^8$ .

The uncertainty related to the stability of the standard during transportation is based on the experience of BIPM with its own transfer standards and is calculated using the following method.

In a comparison which follows the standard protocol for BIPM.EM-K11.b two transfer standards are used.

 $D_1$  and  $D_2$  are the voltage differences between the Josephson standards of the BIPM and the other laboratory, observed during the same comparison for the two transfer

standards. These differences have been calculated for the transfer standards involved in the annual BIPM.EM-K11b comparisons between 2004 and 2007 with NML (Ireland). The absolute value of the difference  $d = (D_1 - D_2)$  gives an estimate on the behavior of the two standards during transportation.

The uncertainty component is then chosen to be the simple mean of these 4 values (2004 to 2007) and is equal to 3 parts in  $10^8$ , in relative terms.

In Table 1, the following elements are listed:

(1) The predicted value  $U_{VNIIM}$  of the Zener, computed using a linear least-squares fit to all of the data from the VNIIM and referenced to the mean date of BIPM's measurements.

(2) The VNIIM Type A uncertainty due to the instability of the Zener is obtained from the following consideration:

Assuming the Zener noise to be white, the Type A uncertainty based on the m=24

measurements carried out at VNIIM would be:  $u_A = \sqrt{\sum_{i=1}^{m} \frac{(U_i - \frac{1}{m} \sum_{i=1}^{m} U_i)^2}{m.(m-1)}} = 46 \text{ nV}.$ 

The value of the Allan variance for an averaging time from 20 s to 1000 s for the transfer standard would give at least 80 nV (Cf. Figure 2), indicating that the main contribution to the noise is flicker noise. This last value is considered as the VNIIM Type A uncertainty.





(3) The uncertainty component arising from the representation of the volt at VNIIM, based on  $K_{J-90}$ .

(4) The BIPM simple mean value of the transfer standard.

(5) The BIPM Type A uncertainty, dominated by flicker noise.

(6) The uncertainty component arising from the representation of the volt at the BIPM.

(7) The uncertainty due to the combined effects of the uncertainties of the pressure and temperature coefficients and to the difference of the mean pressures and temperatures in the participating laboratories.

(8) The uncertainty related to the transfer.

(9) The result of the comparison,  $(U_{VNIIM} - U_{BIPM})$  for the Zener.

(10) The total combined uncertainty.

The final result of the comparison is presented as the difference between the value assigned to a 10 V standard by the VNIIM, at the mean date of the BIPM measurements,  $U_{\text{VNIIM}}$ , and that assigned by the BIPM.

 $U_{\text{VNIIM}} - U_{\text{BIPM}} = -0.22 \ \mu\text{V};$   $u_{\text{c}} = 0.34 \ \mu\text{V}$  on 2007/09/13,

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, the uncertainty of the representation of the volt at VNIIM, both based on  $K_{J-90}$ , and the uncertainty related to the comparison.

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

**Table 1**. Results of the VNIIM (Russia)/BIPM bilateral comparison of 10 V standardsusing one VNIIM Zener travelling standard: reference date 13 September 2007.Uncertainties are 1  $\sigma$  estimates.

	Z_7590806	Contribution /µV
(1)	VNIIM (Russia)(Uz — 10 V)/μV	-4.714
(2)	Type A uncertainty/µV	0.080
(3)	Type B uncertainty/µV	0.006
(4)	<i>ΒΙΡΜ</i> ( <i>U</i> <sub>Z</sub> — 10 V)/μV	-4.497
(5)	Type A uncertainty/µV	0.100
(6)	Type B uncertainty/µV	0.005
(7)	pressure and temperature correction uncertainty/µV	0.080
(8)	Transfer uncertainty/µV	0.300
(9)	$U_{ m VNIIM} - U_{ m BIPM} / \mu  m V$	- 0.218
(10)	combined standard uncertainty/µV	0.336

(*U*<sub>z</sub> — 10 V)

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 maintenance of the volt and the Zener calibration at the VNIIM.

BIPM uncertainty components	Uncertainty/nV
thermal electromotive forces	5
detector / electromagnetic	0.7
interference	
leakage resistance	0.2
frequency	0.2
pressure correction	0.2
temperature correction	80
total	80.2

**Table 2**. Estimated standard Type B uncertainties for the transfer standard measurement with the BIPM equipment.

VNIIM uncertainty components	Uncertainty/nV
thermal electromotive forces	5
detector	3
leakage resistance	0.03
frequency	2
total	6.2

**Table 3**. Estimated standard Type B uncertainties for the transfer standard with the VNIIM equipment.

#### Annex A : Characteristics of the VNIIM JAVS and measurement procedure

• The array step current width is typically 20 μA.

• The waveguide is a oversized circular waveguide (14 mm in diameter) and is inserted in a 1.4 m cryoprobe. The power insertion loss is about 1.5 dB.

• Three pairs of electrical leads are connected to the array. One pair is to supply the current and the two others are dedicated to voltage measurement. The voltage measurement leads have a 10  $\Omega$  series resistance and their leakage resistance is greater than 200 G $\Omega$ .

• The order of magnitude of the thermal EMFs is less than 200 nV.

• The microwave source comprised a back-wave-tube (BWT) oscillator (Model G4-142) from which the power available is 30 mW. The frequency is tuneable in the range from 54 GHz to 79 GHz. During the comparison exercise, the frequency was set to 74.502 800 GHz. The BWT oscillator is phase-locked to an X-band reference signal through a wide-bandwidth control loop assembled at VNIIM. The X-band reference signal is generated by a microwave synthesiser (Model RCh-03) for which the frequency is locked to a 5 MHz reference signal produced by a rubidium frequency standard. The microwave source is electrically isolated from the cryoprobe.

• The DC bias source is adjustable to select the step and can produce a 14 V voltage pulse to set of all junctions to the same polarity. The bias source is referenced to the potential reference and is continuously operating during the measurement.

• The detector is used on its 10 mV range and its internal parameters are set as follow:

- rate 1 pls;
- analog filter off;
- digital filter on;
- counter 14.

All the measurements were carried out without reversing the detector polarity.

#### **Measurement method**

The JVS and the Zener were connected in series opposition and the voltage difference was read with a null detector. Polarity reversal of Zener output voltage and

polarity reversal of JVS ensured that all thermal electromotive forces (EMF) were cancelled out. The typical voltage difference read by the nanovoltmeter was less than 1  $\mu$ V.

The Measurement sequence lasts 1.5 minute per measurement point following the sequence: +/- then -/+, with 280 data readings for each polarity.

### References

[1] V. S. Aleksandrov; A. S. Katkov; G. P. Telitchenko "New State Primary Standard and State Test Scheme for Instruments for Measuring DC Electrical Voltage and Electromotive Force" Measurement Techniques, Volume 45, Issue 3, Mar 2002 Pages: 228-232.

[2] Reymann D., Witt T. J. et al., *Recent Developments in BIPM Voltage Standard Comparison*, IEEE Transactions on instrumentation & measurement, vol. 50, N°2, pp. 206-209, 2001.